PB84-212695

Chlorinated Organic Compounds in Digested, Heat-Conditioned, and Puri fax-Treated Sludges

Camp, Dresser and McKee, Inc., Boston, MA

Prepared for

Municipal Environmental Research Lab.
Cincinnati, OH

Jul 84

U.S. Department of Commerce
National Technical Information Service
NTIS

PB84-212695

EPA-600/2-84-117
July 1984

CHLORINATED ORGANIC COMPOUNDS IN DIGESTED, HEAT-CONDITIONED, AND PURIFAX-TREATED SLUDGES

by

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Contract No. 68-03-2803

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SPRINGFIELD, VA. 22161

TECHNICAL REPORT DATA
(Please read Instructions on the reverse before completing)

1. REPORT NO. EPA-600/2-84-117	2.
3. RECIPIENT'S ACCESSION NO. PB84 212695	
4. TITLE AND SUBTITLE Chlorinated Organic Compounds in Digested, Heat-Conditioned, and Purifax-Treated Sludges	5. REPORT DATE July 1984
	6. PERFORMING ORGANIZATION CODE
7. AUTHOR(S) A. B. Pincince and C. J. Fournier	8. PERFORMING ORGANIZATION REPORT NO.
9. PERFORMING ORGANIZATION NAME AND ADDRESS Camp, Dresser and McKee, Inc. One Center Plaza Boston, Massachusetts 02108	10. PROGRAM ELEMENT NO.
	11. CONTRACT/GRANT NO. 68-03-2803
12. SPONSORING AGENCY NAME AND ADDRESS Municipal Environmental Research Laboratory Office of Research and Development U. S. Environmntal Protection Agency Cincinnati, OH 45268	13. TYPE OF REPORT AND PERIOD COVERED Final
	14. SPONSORING AGENCY CODE EPA/600/14
15. SUPPLEMENTARY NOTES Project Officer: James A. Ryan (513) 684-7653	

16. ABSTRACT

Wastewater sludges were stabilized by Purifax treatment, anaerobic digestion and heat conditioning. The processed sludges from the Purifax process at chlorine dosages normally used in processing wastewater sludges contained 2 to > 14 times the total organic chlorine content of the other processes. Most of the organic chlorine was insoluble and associated with the sludge cake. In analyses for priority pollutants, less than 16 percent of the chlorinated material was recovered.

17. KEY WORDS AND DOCUMENT ANALYSIS		
a. DESCRIPTORS	b. IDENTIFIERS/OPEN ENDED TERMS	c. COSATI Field/Group
18. DISTRIBUTION STATEMENT Release to Public	19. SECURITY CLASS (This Report) Unclassified	21. NO. OF PAGES 106
	20. SECURITY CLASS (This page) Unclassified	22. PRICE

EPA Form 2220-1 (Rev. 4-77) PREVIOUS EDITION IS OBSOLETE

DISCLAIMER

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FOREWORD

The U.S. Environmental Protection Agency was created because of increasing public and government concern about the dangers of pollution to the health and welfare of the American people. Noxious air, foul water, and spoiled land are tragic testimony to the deterioration of our natural environment. The complexity of that environment and the interplay between its components require a concentrated and integrated attack on the problem.

Research and development is that necessary first step in problem solution and it involves defining the problem, measuring its impact, and searching for solutions. The Municipal Environmental Research Laboratory develops new and improved technology and systems for the prevention, treatment, and management of wastewater and solid and hazardous waste pollutant discharges from municipal and community sources, for the preservation and treatment of public drinking water supplies, and to minimize the adverse economic, social, health, and aesthetic effects of pollution. This publication is one of the products of that research; a most vital communications link between the researcher and the user community.

This study was conducted in response to concerns that the Purifax process for sludge stabilization produces unacceptable quantities of chlorinated organics. This study found more chlorinated organics in Purifax-treated sludge than in digested or heat-conditioned sludge. Most of the chlorinated organics were associated with the solid fraction of the sludges. The leachate contained less than 0.2 percent of the total chlorinated organics.

Methods for identifying chlorinated compounds and other priority pollutants need improvement in order to give consistent results and to identify a larger portion of the chlorinated compounds. The fate of chlorinated compounds still needs to be resolved.

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ABSTRACT

There has been concern that the Purifax process produces chlorinated organics that could damage the environment. In order to address these concerns, a study was conducted investigating the effects of sludge stabilization methods on the production of priority pollutants and chlorinated organics. Three stabilization processes -- Purifax treatment, anaerobic digestion, and heat conditioning -- were examined in pilot studies. Cakes and centrates produced by centrifuging sludge samples were analyzed to determine the effects of the processes on concentrations of priority pollutants and chlorinated organics. Leachates produced by the EPA Extraction Procedure were also analyzed.

Wastewater sludges treated by the Purifax process contain higher concentrations of chlorinated organics than raw sludges or sludges that have been heat conditioned or digested. Sludge processed by the Purifax process at chlorine dosages normally used in processing wastewater sludges contains from 2 to more than 14 times the concentration of total organic chlorine in raw sludge. Both insoluble and soluble forms of organic chlorine increase with chlorine dosage.

Most of the chlorinated compounds are associated with the solid fraction of the sludge. About 90 percent of the total organic chlorine is insoluble and about 97 percent or more of the insoluble fraction is associated with the cake. Leachate obtained by the EPA Extraction Procedure (Federal Register, Vol. 43, No. 243, pp. 58956-58957), contained less than 0.2 percent of the organic chlorine in sludge cake after Purifax treatment. These findings are for sludge immediately after processing. It is not known what happens to the concentration of organic chlorine after some time in the environment, for example, after landfilling. This is an area that deserves attention, in order to determine the fate of chlorinated substances.

Methods for identifying chlorinated compounds and other priority pollutants need improvement. The tests for chlorinated compounds identified only a small portion of the total organic chlorine present in sludge or sludge cake. In addition advanced techniques for identifying priority pollutants and chlorinated organics provided inconsistent results.

This report was submitted in fulfillment of Contract No. 68-03-2803 by Camp Dresser & McKee Inc. under the sponsorship of the U.S. Environmental Protection Agency. This report covers the period from December 19, 1979 to May 31, 1982.

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ACKNOWLEDGMENTS

This report "Chlorinated Organic Compounds in Digested, Heat-Conditioned, and Purifax-Treated Sludges" was prepared under the direction of Mr. Paul W. Prendiville, officer-in-charge, and Dr. Albert B. Pincince, project manager. Project engineers were A. Ulvi Gulbey and Christopher J. Fournier. The assistance of Mr. David F. Young and Ms. Diane Chaplick is gratefully acknowledged.

EPA project officer was Dr. James A. Ryan, who provided valuable direction and guidance. Review comments by Dr. Joseph B. Farrell were also appreciated.

SECTION 1

INTRODUCTION

In the Purifax process, chlorine gas is added to sludge, septage, or digester supernatant in order to stabilize and condition the material before dewatering. The process oxidizes the wastes in a closed container at a pressure of about 300 kilopascals for about 10 to 15 minutes. The chlorine dosage is high (about 50,000 mg/kg of dry solids), and the residual concentration is also high (roughly 60 to 400 mg/l). Because of these high concentrations, there has been concern that the process might produce unacceptable concentrations of chlorinated organics.

Previous work has been done to determine concentrations of chlorinated organics in Purifax-treated sludges (Wise, et al., 1978; Stump, et al., 1979),* but the results have never been compared with other stabilization processes and the studies were limited in scope. The objective of this study was to determine the effects of stabilization processes on production of priority pollutants and chlorinated organics. Three stabilization processes -- Purifax treatment, anaerobic digestion, and heat conditioning -- were examined. In these studies a broader range of sludges and controls were used than in earlier studies. Also, additional analytical techniques were used.

In this study, three major tasks were undertaken:

1. Batches of sludges from three wastewater treatment plants -- one processing only domestic water and two receiving some industrial wastes -- were processed in pilot units simulating full-scale operations.

2. Analyses for the priority pollutants prescribed by EPA and for other chlorinated compounds were conducted on the sludges and on their cakes, centrates and leachates. Dewatering was accomplished by centrifuging.

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* Stump, V. L., J. T. O'Connor, and E. S. K. Chian. Evaluation of Purifax Process in Field Operations. Mid-Missouri Engineers, Inc., Columbia, MO 1979. 175 pp.

Wise, R. H., T. A. Pressley, and B. M. Austern. Partial Characterization of Chlorinated Organics in Superchlorinated Septages and Mixed Sludges. EPA-600/2-78-020, U.S. Environmental Protection Agency, Cincinnati, Ohio, 1978. 22 pp.

3. The statistical validity of the test results was examined by conducting tests in which replicate samples were treated by the Purifax process. Results were statistically examined and findings applied to the larger body of results.

Chlorine in the environment can be present in many forms. To avoid confusion, the following nomeclature is used to describe the forms of chlorine:

Chlorine gas
Total chlorine in all forms
Total organic chlorine (soluble and insoluble)
Insoluble organic chlorine
Soluble organic chlorine
Inorganic chlorine (e.g. HOCl, Cl-, and Chloramines)
Chloride ion
Residual chlorine (total chlorine minus chloride)

SECTION 2

CONCLUSIONS AND RECOMMENDATIONS

Wastewater sludges treated by the Purifax process contain higher concentrations of chlorinated organics than raw sludges or sludges that have been heat conditioned or digested. Sludge processed by the Purifax process at chlorine dosages normally used in processing wastewater sludges contains from 2 to more than 14 times the concentration of total organic chlorine in raw sludge. Both insoluble and soluble forms of organic chlorine increase with chlorine dosage. The differences in concentrations of insoluble organic chlorine in the cake between Purifax samples and samples of feed, digester seed, heat-conditioned sludge, and digested sludge are statistically significant at levels exceeding 77 percent.

Most of the chlorinated compounds are associated with the solid fraction of the sludge. About 90 percent of the total organic chlorine is insoluble and about 97 percent or more of the insoluble fraction is associated with the cake. Leachate obtained by the EPA Extraction Procedure (Federal Register, Vol. 43, No. 243, pp. 58956-58957), contained less than 0.2 percent of the organic chlorine in sludge cake after Purifax treatment. These findings are for sludge immediately after processing. It is not known what happens to the concentration of organic chlorine after some time in the environment, for example, after landfilling. This is an area that deserves attention, in order to determine the fate of chlorinated substances.

Methods for identifying chlorinated compounds and other priority pollutants need impovement. The tests for chlorinated compounds identified only a small portion of the total organic chlorine present in sludge or sludge cake. In addition advanced techniques for identifying priority pollutants and chlorinated organics provided inconsistent results.

SECTION 3

SLUDGE PROCESSING TESTS

SELECTION OF WASTEWATER TREATMENT SLUDGES

To allow for the variability of sludges in plants receiving different types of wastewater, we tested sludges from three wastewater treatment plants -- Amherst, MA; Willimantic, CT; and Pittsfield, MA. The Amherst plant serves a residential area that has developed around several college campuses. The Willimantic plant receives flow from some 20 industries. This flow constitutes about 25 percent of the total flow for the plant. Industrial processes include dyeing of cotton and synthetics, production of woolen fabrics, and poultry processing. About 5 percent of the total flow at the Pittsfield plant comes from two industries: General Electric and the Crane Paper Company. The General Electric plant discharges pretreated wastes containing phenolic substances. The Crane Paper Company's main discharge consists of liquids from a pulp digestion process.

PILOT PLANT PROCESSES

The stabilization operations were simulated by using batch pilot-testing equipment. The pilot heat-treatment equipment and digesters were run at Camp Dresser & McKee's (CDM's) Boston laboratories. The pilot Purifax unit was operated at the West Warwick, RI laboratory of BIF, the Purifax equipment manufacturer. Basic methods are capsulized below; see Appendix A for details.

Anaerobic Digesters

Batch digestion operations were conducted in specially adapted 10-liter vessels. A sample of previously digested sludge was used to seed the digester to start the tests and to obtain proper performance. Sludge from the Pittsfield plant was seeded with a sludge from the Pittsfield digesters. On tests from the Willimantic and Amherst sludges, seed from the treatment plant in Ansonia, CT was used. (The Ansonia plant processes domestic wastes only.)

The feed material added to the batch digesters included mixtures of raw sludge and digested sludge seed in two ratios of raw sludge to seed by volume, 25/75 and 50/50.

Digestion temperature was maintained at 35°C and the digester contents were continually mixed with digester gas. The tests lasted 28 days. Gas production was very low at that time and changes in total volatile solids were minimal. Also, the total volatile solids in the sludge approached concentration in the digester seed.

Heat Treatment

Bench-scale heat-conditioning tests were conducted in a 1-liter autoclave unit. This unit was used as a batch heat-conditioning system in which approximately 0.8 liters of sludge were mixed and heated for 30 minutes to 180°C at 970 kPa. (Full-scale heat-conditioning units operate at 120 to 220°C and 1700 to 2000 kPa for 20 to 30 minutes.)

Purifax Pilot Tests

Tests for Purifax treatment of sludge were performed by BIF personnel using a batch unit at their West Warwick, RI manufacturing plant. A senior engineer from CDM observed the operation.

The pilot plant tests were conducted to provide sludges having what could be termed high, medium, and low chlorine residuals, as compared with operation at full-scale Purifax processing plants which would correspond to the medium chlorine residual. Medium residuals were defined as those between 150 and 200 mg/l; high residuals between 280 and 350 mg/l; and low residuals were from 60 to 120 mg/l. The body of this report evaluates results for medium residuals. Data on high and low residuals are in the appendices.

PROCEDURES

Care was taken in sludge handling to ensure that sludge samples collected represented their respective plants and that testing portions were uniform. (Detailed handling procedures are discussed in Appendix B.) The samples were cooled after collection and carefully stirred before they were divided into testing portions. After digestion, heat conditioning, and Purifax tests had been conducted, the samples were centrifuged to produce a cake and a centrate. These products were then analyzed for chlorine compounds. Analytical methods used to conduct these tests are described in Appendix C; Appendix D details the quality assurance program followed for organic priority pollutants.

SECTION 4

RESULTS AND DISCUSSION

EFFECT OF SLUDGE PROCESS ON CHLORINE CONTENT

Figure 1 is a bar chart showing the concentration of total organic chlorine in sludges from the three plants before and after treatment. The Purifax-treated sludges are the only ones showing substantial increases in total organic chlorine after processing. Except for the results for heat-conditioned sludge from Pittsfield, which we think represents an error, the concentration of total organic chlorine in Purifax treated sludge is from 2 to more than 14 times the concentration in raw sludge or in heat conditioned or digested sludge. These ratios are for a chlorine residual of about 170 mg/l (a "medium" residual), which occurred from a dosage of about 1500 mg/l. The dosage and residual are comparable to those used at Purifax plants. (See Table 1 for data on total organic chlorine.)

In order to assess the validity of the chlorine data, recoveries of total chlorine in the samples that were digested and heat-conditioned were evaluated. The sum of organic and inorganic chlorine in these samples should not vary from the amount determined in the feed sludge, since no chlorine is added in the processes. The recoveries of total chlorine are presented in Table 2. Ratios of total chlorine in treated sludges to total chlorine in the sludge charged to digestion or heat conditioning ranged from 0.66 to 1.27.

As discussed in Appendix F, the validity of these results were further examined in studies conducted to determine the statistical significance of the analyses. In 26 out of 27 samples, the probability that the concentration of insoluble organic chlorine is the same in a sludge cake after treatment by the Purifax process as after heat treatment or anaerobic digestion is less than 22.5 percent, using a conservative statistical model (the Tchebychev inequality). With a less-conservative model, the probability is less than 16 percent. The exception was on sludge from the Pittsfield plant where the cake from low-dosage Purifax treatment contained less insoluble organic chlorine than the cake from heat conditioning.

TABLE 1. EFFECT OF STABILIZATION PROCESS ON CONTENT OF TOTAL ORGANIC CHLORINE

Type of Sludge	Total Organic Chlorine (mg/l)* Amherst Sludge	Pittsfield Sludge	Willimantic Sludge
Raw	38	106	29
Digested #	24	25	16
Heat Conditioned	22	136**	8
Purifax treated ##	129	268	115
Seed used for digester	74	69	21

* Averages of available data.
\# Digested mixture of equal volumes of raw sludge and digested sludge seed.
Sample with "medium" chlorine residual.
** Atypical result, probably an error.

TABLE 2. PERCENT RECOVERY OF TOTAL CHLORIDES

Type of Sludge	Pittsfield	Amherst	Willimantic
Digested*	66	79	127
Heat Conditioned	119	101	119

* Digested mixture of equal volumes of raw sudge and digested sludge seed.

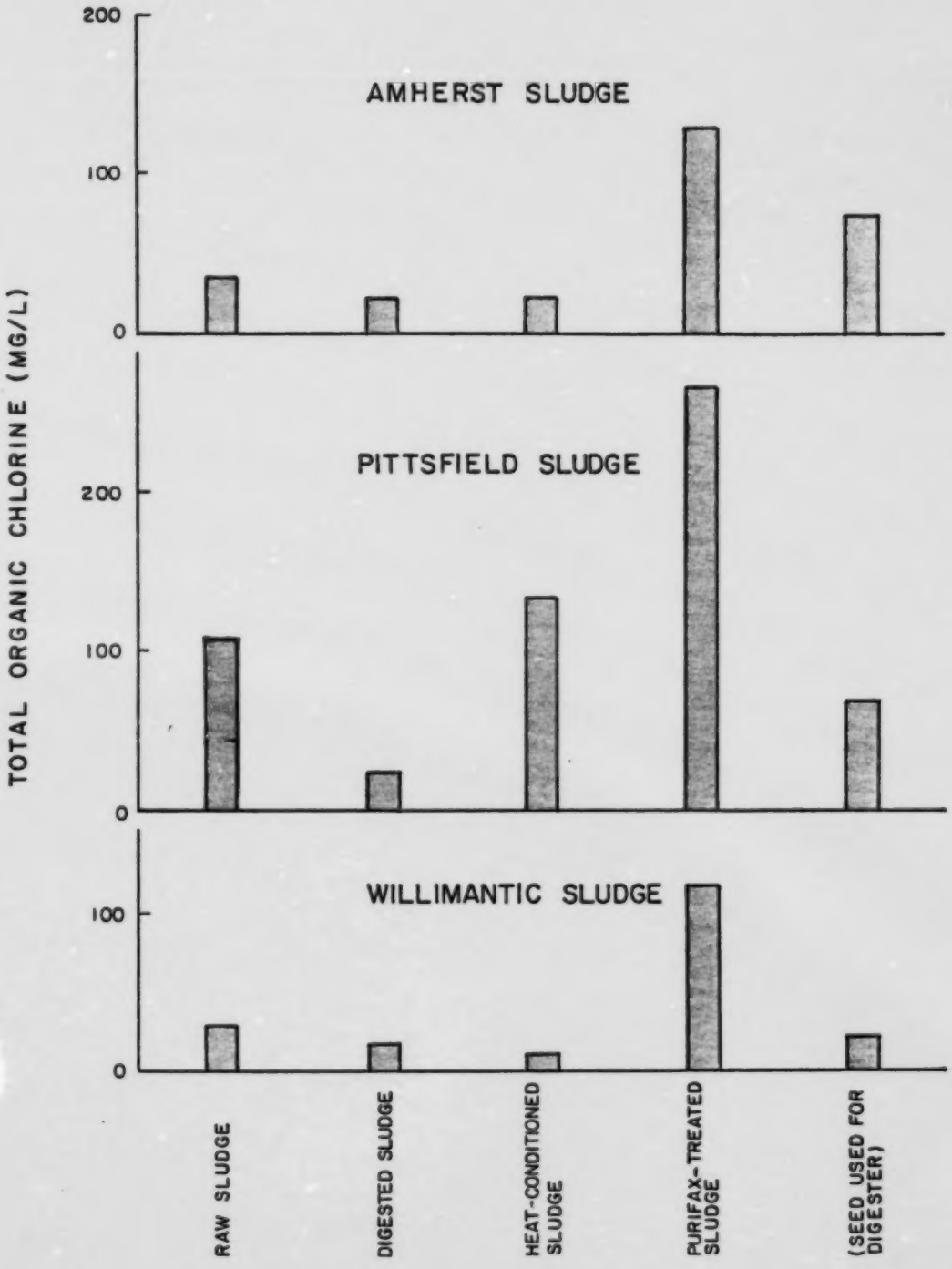


FIGURE 1. EFFECT OF STABILIZATION PROCESS ON CONTENT OF TOTAL ORGANIC CHLORINE

EFFECT OF CHLORINE DOSAGE AND CHLORINE RESIDUAL ON ORGANIC CHLORINE CONTENT

As shown on Figures 2 and 3, the concentration of soluble and insoluble organic chlorine increases with increased chlorine residual or chlorine dosage. There is scatter to the data for all four combinations, but a statistically significant line of fit can be drawn through the data; as indicated. Coefficients of determination (r^2) range from 0.18 to 0.32. For the number of samples in each case, the trends are significant at the levels indicated in the figures (1.5 to 8.1 percent).

DISTRIBUTION OF CHLORINATED COMPOUNDS IN SAMPLES AND LEACHATES

The tests showed that most of the organic chlorine in all tested sludges is insoluble, and that most of the total organic chlorine remains in the cake. Of the portion in the cake, only a small portion is leachable.

Table 3 shows the concentration of soluble and insoluble organic chlorine in sludges from the three plants after processing. More than 94 percent of the total organic chlorine is insoluble. For Purifax-treated sludge, more than 97 percent of the organic chlorine is insoluble.

Since most of the organic chlorine is insoluble, it is not surprising to find that little of the organic chlorine ends up in the centrate after centrifuging. Table 4 shows that less than 3 percent of the total organic chlorine that is present in processed sludge is in the centrate. In the Purifax-treated samples, only 1.4 percent of the chlorine was in the centrate.

TABLE 3. ORGANIC CHLORINE IN SLUDGES*
(mg/l)

	Amherst		Pittsfield		Willimantic	
	Soluble	Insoluble	Soluble	Insoluble	Soluble	Insoluble
Raw	0.36	37.3	0.08	105.7	0.02	28.6
Digested #	0.63	23.7	1.4	23.2	0.016	15.9
Heat conditioned	0.8	21.4	0.07	136.3	0.02	8.4
Purifax-treated ##	1.55	127.6	1.3	266.3	2.1	112.7
Seed for digester	ND	74.1	ND	68.5	0.51	20.6

* Organic chlorine in cake plus centrate (mg/l). Averages of available data.
\# Digested mixture of equal volumes of raw sludge and digested sludge seed.
Sample with "medium" chlorine residual.
ND Means none detected.

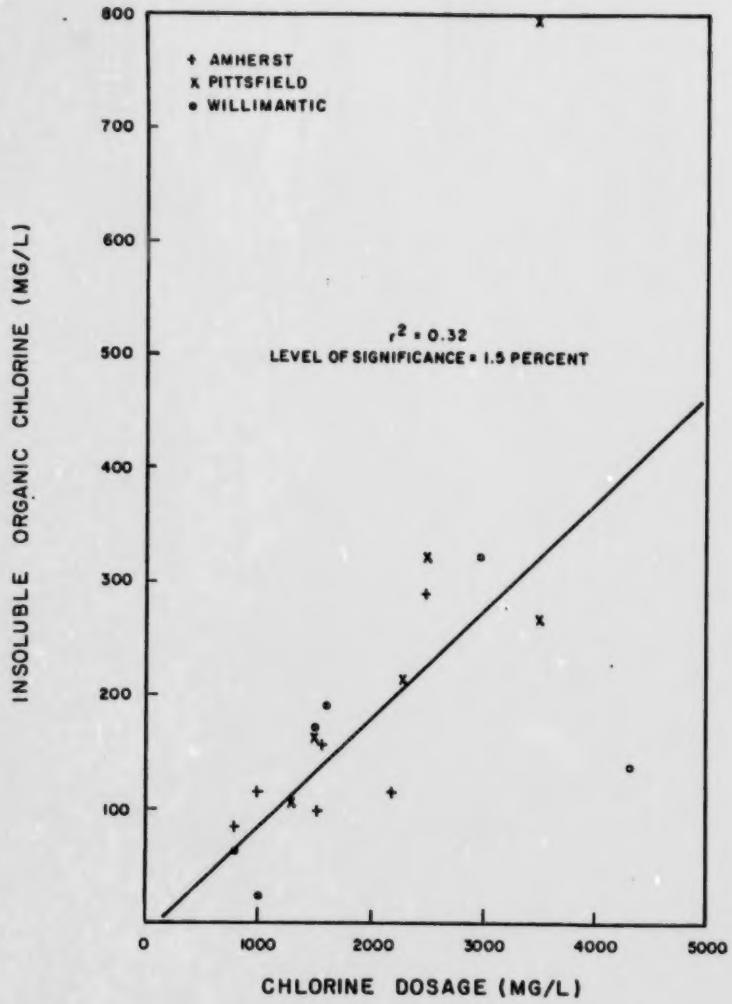
TABLE 4. PERCENTAGE OF TOTAL ORGANIC CHLORINE IN CENTRATE**

Type of Sludge	Centrate*
Raw	0.5
Digested #	1.1
Heat conditioned	2.8
Purifax treated ##	1.4

* Averages of available data for sludge from Amherst plant.
\# Digested mixture of equal volumes of raw sludges and digested sludge as seed.
Sample with "medium" chlorine dosage.
** $(100)\ \frac{(\text{TOCl in centrate})}{(\text{TOCl in centrate \& TOCl in Cake})}$

Table 5 shows the concentrations of total organic chlorine in leachate and the percentage of total organic chlorine in the cake that found its way into the leachate. For all samples, the concentration of total organic chlorine in the leachate amounted to less than 0.1 mg/l, and less than 1.5 percent of the total organic chlorine ended up in the leachate. In terms of the soluble organic chlorine, only one-third of the soluble chlorine associated with the cake finds its way into the leachate. One would expect that all of the soluble organic chlorine in the cake would be extracted in EPA's extraction test. However, the conditions to determine soluble organic chlorine in cake (extraction with hexane and diethylether) might extract more organic material than the EPA extraction procedure with an acetic acid solution.

Clearly the bulk of the organic chlorine remains with the sludge cake. Therefore, the fate of the cake and the decomposition of the organic chlorine need to be determined in order to fully evaluate the impact of sludge treatment on release of organic chlorine to the environment.



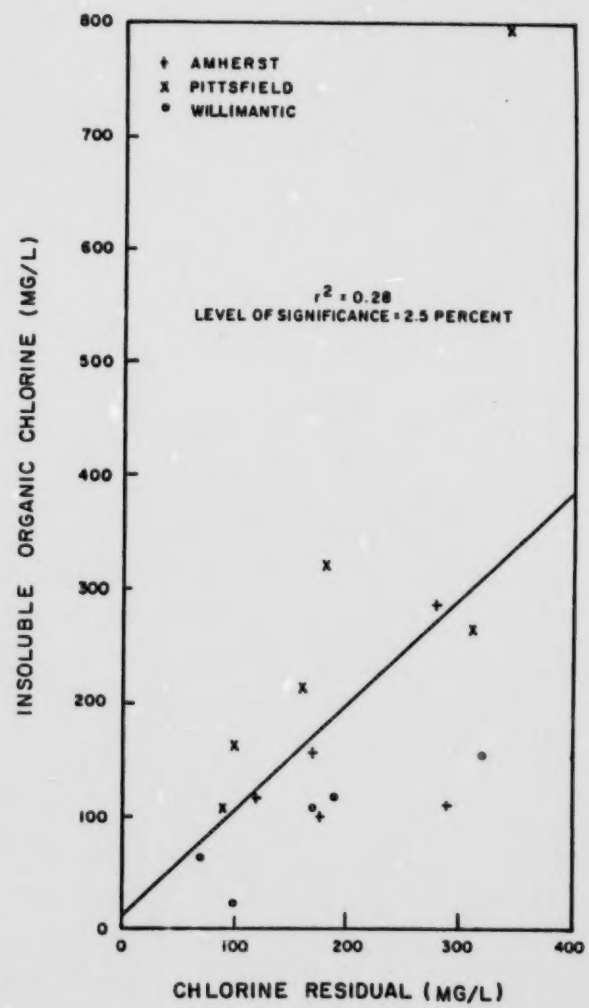


FIGURE 2. EFFECT OF CHLORINE DOSAGE AND RESIDUAL ON INSOLUBLE ORGANIC CHLORINE

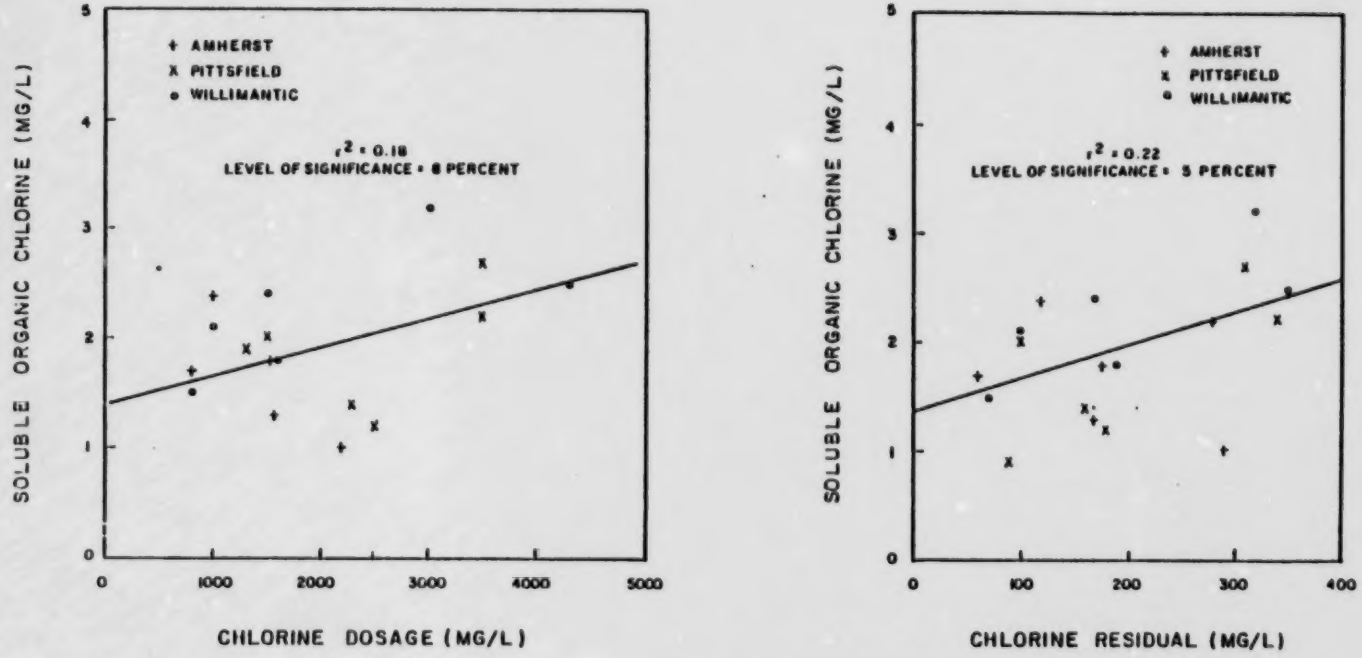


FIGURE 3. EFFECT OF CHLORINE DOSAGE AND RESIDUAL ON SOLUBLE ORGANIC CHLORINE

TABLE 5. TOTAL ORGANIC CHLORINE IN LEACHATE FROM AMHERST SLUDGE*

Type of Sludge	Concentration of Organic Chlorine in Leachate (mg/l)	Percentage of Organic Chlorine in Leachate to Organic Chlorine in Cake	
		Soluble	Total
Raw	0.02	32	0.2
Heat conditioned	0.10	69	1.5
Digested **	0.017	17	0.3
Purifax treated	0.07	34	0.2

* As specified in Federal Register Vol. 43, No. 243, pp. 58956-58957. Averages of available data. Total organic chlorine is all soluble.

** Digested mixture of 25 percent raw sludge and 75 percent digested sludge serving as feed.

TESTS TO DETERMINE SPECIFIC CHLORINATED COMPOUNDS

Analyses were conducted to identify the chlorinated compounds present in the raw sludge and in the sludge products from the Amherst wastewater treatment plant. The analyses included those for organic priority pollutants and a new technique developed by the Research Triangle Institute (RTI) to identify chlorinated organics.

Data for the priority pollutants are presented in Table 6 for sludge and in Table 7 for sludge cake. The volatile constituents (101 through 127) were analyzed only in the sludge because they would have been removed when the sludge cake was prepared. Of these compounds, chloroethane (105) and carbon tetrachloride (116) are present in Purifax sludge and not in any others.

Chloroform (113) is present in the feed and in the Purifax samples, but the concentration in the Purifax samples is an order of magnitude higher.

TABLE 6. COMPARISON OF CHLORINATED PRIORITY POLLUTANTS IN AMHERST SLUDGE (µg/l)

Analysis		Seed	Feed		Heat Treated	Digested	Purifex	
		(510)*	(508)	(509)	(511)	(514)	(506)	(507)
101	Chloromethane	3	-	-	-	-	19	48
102	Dichlorodifluoromethane	-	-	-	21	-	-	-
105	Chloroethane	-	-	-	-	-	21	32
106	Methylene Chloride	-	-	-	-	99	-	-
112	Trans-1,2-Dichloroethylene	3	-	-	-	-	-	-
113	Chloroform	-	10	20	-	-	150	160
115	1,1,1-Trichloroethane	-	1	2	-	32	3	2
116	Carbon Tetrachloride	-	-	-	-	-	170	226
120	Trichlorethylene	-	1	2	-	-	1	1
127	1,1,2,-Tetrachloroethylene	-	2	6	-	-	4	7
201	2-Chlorophenol	-	-	-	-	-	P	P
205	2,4-Dichlorophenol	-	P	-	-	-	-	11
206	2,4,6-Trichlorophenol	-	-	-	-	-	20	26
210	Pentachlorophenol	-	P	P	-	-	P	P
301-3	Dichlorobenzene	87	11	13	12	-	12	10
312	1,2,4-Trichlorobenzene	-	-	-	-	-	-	-
402	Gamma-BHC	-	-	-	-	-	-	-
403	Heptachlor	-	-	-	-	-	-	-
404	Beta-BHC	-	-	-	-	-	-	-
409	DDE	-	-	-	-	-	-	-
410	Dieldrin	-	-	-	-	-	-	-
412	DDD	-	12	-	-	-	-	-
414	DDT	-	190	-	-	-	-	-
417	Chlordane	-	A	A	A	-	B	B
419	PCB-1221	-	-	A	-	-	C	C
420	PCB-1232	-	-	A	-	-	-	-
421	PCB-1242	-	-	-	-	A	-	-
422	PCB-1248	B	C	-	-	-	B	C
423	PCB-1254	B	D	A	-	-	A	B
424	PCB-1260	-	-	-	-	-	-	B
425	PCB-1016	-	-	A	-	-	-	-

* Sample numbers.
\- Not detected.
P Detected but below reporting limit.
A 10 to 100 µg/l.
B 100 to 1000 µg/l.
C 1000 to 5000 µg/l.
D 5000 to 10,000 µg/l.

TABLE 7. COMPARISON OF CHLORINATED PRIORITY POLLUTANTS IN AMHERST SLUDGE CAKE (µg/kg)

Analysis		Seed	Feed		Heat Treated	Digested	Purifax	
		(540)*	(502)	(503)	(515)	(505)	(500)	(501)
201	2-Chlorophenol	-	-	-	-	-	-	-
205	2,4-Dichlorophenol	-	-	P	-	-	P	P
206	2,4,6-Trichlorophenol	-	-	P	-	-	P	130
210	Pentachlorophenol	-	P	120	-	-	P	P
301-3	Dichlorobenzene	360	70	70	-	130	-	170
312	1,2,4-Trichlorobenzene	-	-	-	-	150	-	-
402	Gamma-BHC							
403	Heptachlor	39	-	-	-	-	-	-
404	Beta-BHC	-	-	36	-	-	-	-
409	DDE	-	-	-	-	26	-	-
410	Dieldrin	-	28	-	-	-	-	-
412	DDD	50	50	-	-	-	-	-
414	DDT	-	-	-	-	-	-	-
417	Chlordane	C	A	A	-	-	-	-
419	PCB-1221	-	-	B	-	-	-	-
420	PCB-1232	-	A	A	-	-	-	-
421	PCB-1242	-	-	-	-	-	-	-
422	PCB-1248	D	C	-	-	-	C	C
423	PCB-1254	D	-	B	-	B	B	B
424	PCB-1260	-	-	-	-	-	-	-
425	PCB-1016	-	-	A	-	-	-	-

* Sample numbers.
\- Not detected.
P Detected but below reporting limit.
A 10 to 100 µ/kg in the sludge cake.
B 100 to 1000 µ/kg in the sludge cake.
C 1000 to 5000 µ/kg in the sludge cake.
D 5000 to 10,000 µ/kg in the sludge cake.

Data for many other constituents appear inconclusive, however. For example, the concentration of 2,4,6-trichlorophenol is higher in Purifax samples of cake than in other cake samples, but there are some other compounds (e.g., DDD (412)) that were present in higher concentrations in other samples.

These analyses identified only a portion of the chlorinated organics in the sludge, as illustrated in Table 8 for two sludges -- Feed "A" and Purifax "A". The table was prepared by comparing the chlorine content of identified compounds with the total chlorine. Comparison is hindered because results of some analyses were reported only in ranges. If the lower value of the ranges is used, then about 6.6 percent of the total organic chlorine in feed sludge is identified. With the upper range, about 16 percent is identified. With Purifax-treated sludge, the recovery is between about 0.7 and 2.6 percent. Identifications for cake are much less.

To confirm testing for priority pollutants and to extend the number of compounds identified, the sludge samples were subjected to a new method of analyses using ionization/gas chromatography/mass spectrometry techniques. To do this, a portion of each extract produced in the analysis for priority pollutants were analyzed by the Research Triangle Institute (RTI). The RTI tests indicated the presence of chlorinated material in all the sludge types analyzed; however, except for PCB (which was found in all types) and chlordane (which was identified in cake from heat-treated sludge, specific compounds could be identified only in the samples that received Purifax treatment (Table 9). The greatest variety of chlorinated organics was found in the sludge, but organics were identified in the cake and leachate. No chlorinated organics were identified in the centrate. The RTI tests did not confirm many of the chlorinated priority pollutants, but RTI's data analyses indicated that chlorinated compounds with properties similar to some chlorinated priority pollutants were present, although their identity was different.

Comments on Analyses for Chlorinated Hydrocarbons

PCB's -- Of the 14 samples for which priority pollutant testing was done:

- 10 were found to contain PCB's in both acidic or basic extracts by RTI;
- 3 samples were confirmed PCB presence for the acidic extracts by RTI (although not for basic extracts);
- 1 sample only, a Purifax sludge cake, resulted in a completely negative test by RTI.

TABLE 8. PERCENT OF TOTAL ORGANIC CHLORINE IDENTIFIED IN AMHERST SLUDGE*

	Sludge Samples		Cake Samples	
	Feed "A"	Purifax "A"	Feed "A"	Purifax "A"
Using lower limits of results of analyses reporting a range.	6.6	0.7	0.03	0.02
Using upper limits of results of analyses reporting a range.	16	2.6	0.1	0.09

* Data for identified compounds comes from Tables 6 and 7. Data for total organic chlorine concentration comes from Table E3.

TABLE 9. CHLORINATED ORGANICS IDENTIFIED BY RTI ANALYSES OF PURIFAX-TREATED SLUDGE

Sample	Chlorinated Compound
Sludge	PCB. Also chloroacetophenone or chloropropylbenzene in one of two samples.
Cake	Tetrachlorobutane, trichloromethylpropene, and chloroacetophenone or chloropropylbenzene. Also PCB and chlorophenol in one of two samples.
Centrate	PCB
Leachate	Chloroacetophone or chloropropylbenzene, and PCB.

Discrepancies that were evidenced are probably attributable to the complexity of the material being examined. There is no evidence regarding the relative efficiency of extraction for the acid or basic solution; however, differences in adsorbability or particle size could affect extraction from one solution more than from the other. Also, there may have been differences in background extractibles or in cleanup problems for the two types of extract.

In any event, it seems safe to say that there are PCB's in most, if not all of these samples, but there are no identifiably consistent differences based on method of sludge treatment. For example, the highest concentrations of PCB's were found in samples 504 (Seed Cake) and 508 (Feed Sludge).

Chlorophenols -- Since the P symbols in Tables 6 and 7 do not represent positive identifications, few, if any of the results on the different chlorophenols have much significance. The positive results on trichlorophenols for samples 501 (Purifax Cake) and 506 (Purifax Sludge) are not confirmed by priority pollutant testing on sample 500 (Purifax Cake) or by any RTI results. Similarly, the strongly positive result on Sample 503 (Feed Cake) is not confirmed on parallel samples or on Purifaxed or other treated samples or by RTI. No conclusions seem possible.

Chlorobenzenes -- The biggest discrepancy between the priority pollutant and RTI results occur in the reports on dichlorobenzes. Measurable quantities were found in almost all samples tested for priority pollutants; none were found by RTI. Dichlorobenzenes are likely to occur widely in sewages and sludges because of household uses, but even the priority pollutant data showed no consistent trends between feed and treated sludges. It appears as if these substances pass through the processing unchanged. Results on other chlorobenzenes are sporadic and show no relationship to treatment.

Conclusion regarding analyses -- Testing procedures need refinement to assure their precision and accuracy for chlorinated compounds in complex materials like sludge.

Pesticides -- The only pesticide found in more than one sample was chlordane. Priority pollutant test found chlordane in six different samples, but none were confirmed by RTI. Four of the positive results were on untreated samples. The other pesticides that were found in one sample each, Dieldrin, DDD, and DDE, were not found in Purifax-treated samples.

APPENDIX A

PILOT PLANT PROCESSES

ANAEROBIC DIGESTERS

Digested sludge from the Pittsfield plant and from the wastewater treatment plant in Ansonia, CT served as seed to start digestion. The digested sludge from Pittsfield was used to seed the digestion of the raw sludge sample obtained from the Pittsfield plant. The digester seed from Ansonia was used to start digestion of raw sludges from the Willimantic and Amherst plants. The Ansonia plant has a capacity of 11,355 cu m/d and processes domestic wastes only.

Duplicate batch digesters were filled with sludges mixed in two volumetric ratios of raw sludge to digested sludge seed: 25/75, 50/50. (A single digester with digested sludge only served as a control.)

Each digester consisted of a specially fabricated pyrex vessel with a capacity of 9.5 liters, a gas collection and measuring system, and a gas recirculation system (See Figure A1). The digesters were fitted with a gas sampling port at the top and a sludge sampling port located 5 cm from the bottom of the bottle.

Continuous mixing was accomplished using a 5-liter/minute aquarium pump modified so as to be sealed from the atmosphere. Digester gas withdrawn from the top of the digester passed through a condensate trap before reaching the recirculation pump. The gas was dispersed near the bottom of the bottle through a glass diffuser. The induced turbulence kept the contents mixed.

Gas production rate was measured by noting the volume of liquid displaced in the gas collector system (Figure A1). An acidified salt solution was used in the measuring system (and in the condensate trap) to prevent absorption of carbon dioxide. A pressure of approximately 30 cm of water was maintained by the elevation of the gas measuring system.

Temperature was maintained at 35°C in a water bath surrounding the digesters. Two wood tanks lined with polyethylene were constructed to hold five digesters each. The temperature was maintained by placing a water-bed heater under the polyethylene. In addition, room temperature was maintained near 35°C to minimize condensation in the gas system.

At the start of digestion, approximately 3600 mg/l of sodium bicarbonate were added to serve as buffer against the increasing volatile acids. When necessary, more sodium bicarbonate was added to maintain acceptable pH,

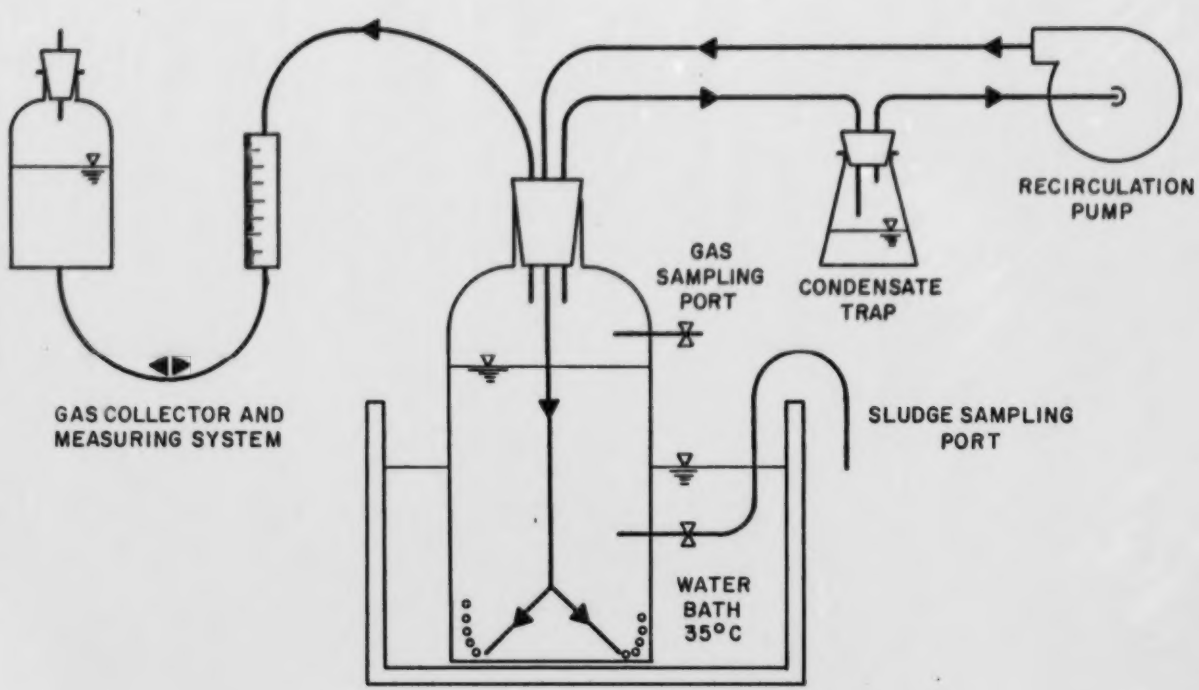


FIGURE A I. SCHEMATIC DIAGRAM OF PILOT-SCALE ANAEROBIC DIGESTER
(9.5 LITER CAPACITY)

volatile acid, and alkalinity levels in the digester.

The degree of digestion was monitored weekly by analyses for total solids, volatile solids, chemical oxygen demand, and ammonia nitrogen. The results of these tests were then compared to baseline samples taken when the digesters were initially set up. In addition, pH and methane production were monitored daily.

The digesters were allowed to run a minimum of 28 days. By that time, gas production had virtually ceased (Figure A2).

HEAT TREATMENT

Bench-scale heat-conditioning tests were conducted in a one-liter-capacity autoclave unit manufactured by Autoclave Engineers, Erie, PA, and supplied by the EPA. This unit was used as a batch heat-conditioning system in which approximately 0.8 liter of sludge was continuously mixed and heated to 180°C at 970 kPa for 30 minutes. (Full-scale heat-conditioning units operate at 120 to 200°C and 1700 to 2000 kPa for 20 to 30 minutes).

At the end of the 30-minute retention time, heating was discontinued and the unit's cooling water system was run for about 15 minutes. The conditioned sludge was then removed by suction at the completion of the cooling cycle (at a temperature of about 50°C). Following each test, the autoclave well and mixer were rinsed with distilled water. Typical test duration was 2 hours, including autoclave preparation, heating, retention at 180°C, cooling cycle, sludge removal, and rinsing.

PURIFAX PILOT TESTS

Tests for chlorination of sludge were performed with a Purifax Laboratory Test Unit. The unit is a miniaturized batch-type system designed for field and laboratory evaluation of waste sludge treatment by the Purifax process. The tests were done by BIF personnel at West Warwick, RI, under the supervision of a senior engineer from CDM.

The pilot tests were conducted to provide sludge products having high, medium, and low chlorine residuals. Because residuals could not be predicted, tests were run using varying dosages until two samples of each concentration range were produced. High residuals were defined as falling between 280 and 350 mg/l; medium residuals ranged from 150 to 200 mg/l; and low residuals were 60 to 120 mg/l. The test unit is shown schematically in Figure A3.

Sample Preparation -- Since the laboratory test unit is a miniaturized Purifax unit, raw sludge to be processed was macerated in a blender, to decrease particle size.

Operation of the Test Unit -- After connection of all the components, a check was made for gas-tight integrity of the equipment. The operational procedure was as follows:

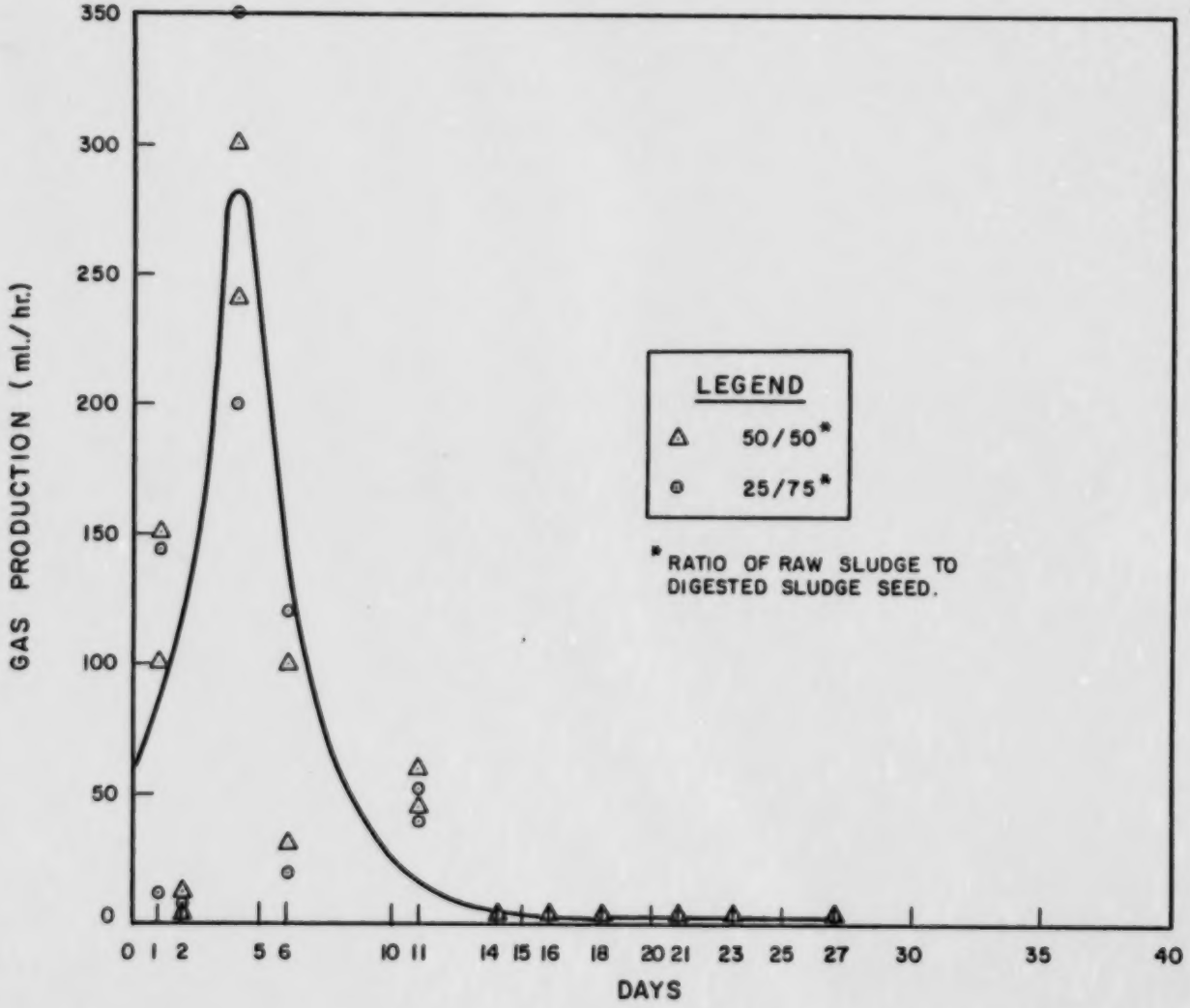


FIGURE A2. GAS PRODUCTION

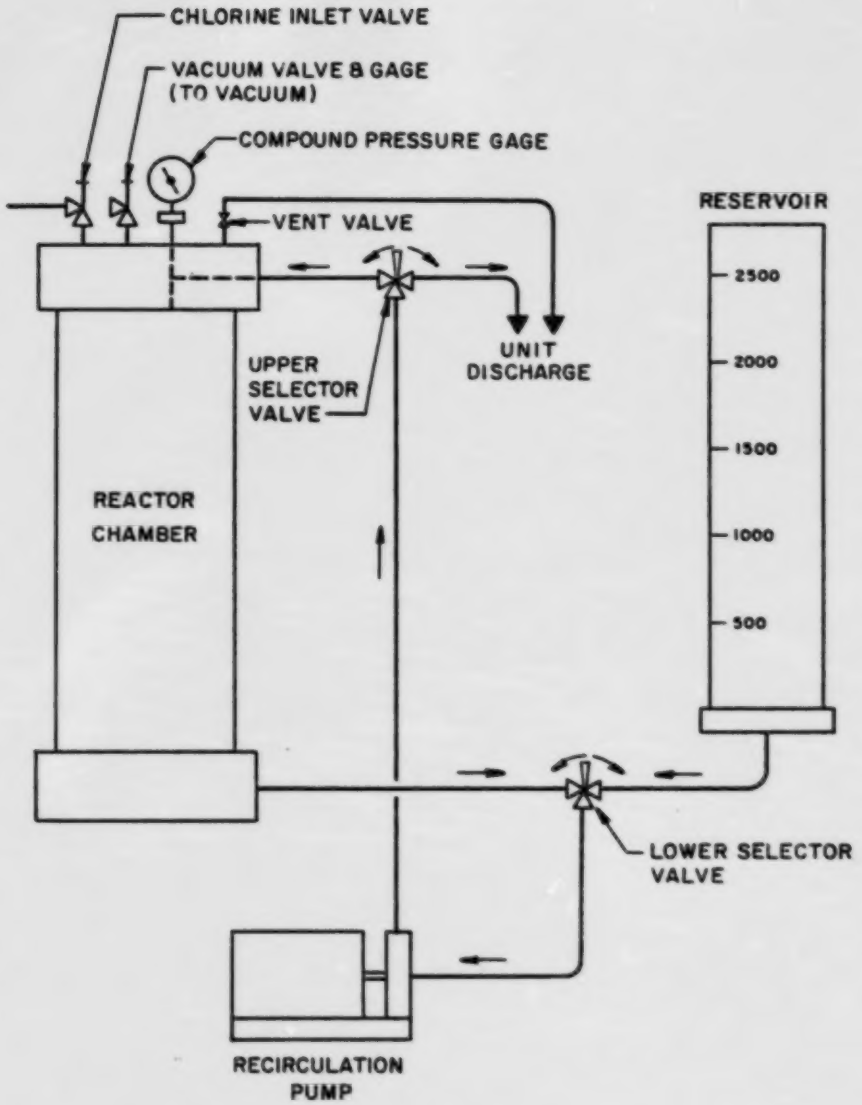


FIGURE A3. PURIFAX LABORATORY TEST UNIT

a. Pour 2,500 ml of macerated sludge into the reservoir.

b. Pump a predetermined volume of sludge from the reservoir into the reaction chamber. (The volume is selected to obtain the desired chlorine dosage).

c. Shut off the pump.

d. Apply a 660-mm Hg vacuum to the reaction chamber.

e. Introduce chlorine to the reactor chamber until the pressure is 5 psig. Pump from the reservoir into the reactor chamber. The sludge level will start to drop in the reservoir. The reactor chamber pressure will rise.

f. When the reactor chamber pressure reaches 35 psig, circulate flow through the reactor chamber. As the chlorine gas combines with the sludge, the reactor chamber pressure will slowly start to drop. The process of pumping in more sludge must be repeated until the reactor chamber compound pressure becomes stable at approximately 30 psi. (About 100 to 250 ml of sludge remains in the reservoir.) When the reactor chamber pressure stabilizes, allow the unit to operate for a total of 10 minutes.

g. Discharge the processed sludge. (Open the vent valve when the pressure in the reaction chamber falls below zero.)

h. Flush the unit and wash with tap water after each use.

i. Measure chlorine residual with a Hach chlorine test kit. Since free chlorine concentrations in the sample diminish gradually, time of chlorine residual reading is critical when comparing the results.

Purifax pilot tests were carried out on sludge samples from the three plants. See the test data shown on Tables A1 through A3. In addition, reliability tests were run to test the statistical significance of findings. (See Appendix E.)

For tests on the Amherst sludge, a field blank (deionized distilled water) passed through (1) the blenders; (2) the galvanized metal container which was used for sludge mixing; (3) the beaker and the glassware, which were used for handling sludge samples; and (4) the Purifax unit. The field blank sample was then sealed off and preserved with ice for subsequent laboratory tests. These blanks were used in the quality control programs.

An attempt was made to determine chlorine residuals in the Purifax-tested sludge with chemical titration by using PAO reagent to check the accuracy of results obtained with the Hach kit. This method, however, was abandoned after several tests because it proved more time consuming and more difficult to detect end point readings when compared to the Hach kit.

TABLE A1. DATA FOR PURIFAX TESTS ON PITTSFIELD SLUDGE

Run No.	Time	Raw Sludge pH	Raw Sludge % Solids	Raw Sludge Color	Raw Sludge °C	Chlorine Application (mg/l) Dosage*	Residual	Demand	Purifax Treated Sludge Treat. Period (min)	pH	Color	°C	Identification+
1	10:30 am	6.2	4.4	Black	9	3,300	280	3,020	10	2.2	Dark Brown	18	The equipment was flushed with sludge prior to the test
2	11:20	6.2	4.5	Black	9	2,500	180	2,320	10	2.8	Dark Brown	20	Medium "A"
3	11:55	6.2	4.5	Black	10	1,500	100	1,300	10	3.5	Dark Brown	19	Low "A"
4	1:20 pm	5.8	4.5	Black	12	3,500	310	3,190	10	2.1	Dark Brown	21	High "B"
5	1:55	5.8	4.5	Black	13	3,500	340	3,160	10	1.6	Dark Brown	22	High "A"
6	2:30	5.8	4.5	Black	14	2,300	160	2,140	10	2.9	Dark Brown	21	Medium "B"
7	3:00	5.75	4.5	Black	14	1,300	90	1,210	10	3.8	Dark Brown	21	Low "B"

*From BIF chart (based on initial quantity of sludge pumped into reaction chamber).
+Sample identification used in Appendix E.

TABLE A2. DATA FOR PURIFAX TESTS ON AMHERST SLUDGE

Run No.	Time	Raw Sludge pH	% Solids	Color	℄	Chlorine Application (mg/l) Dosage*	Residual	Demand	Purifax Treated Sludge Treat. Period (min)	pH	Color	°F	Identification and Comments+
1	9:30 am	6.1	3.1	Dark Grey	12	2,200	290	1,910	10	2.4	Light Brown	-	High "A"
2	10:00	6.1	3.0	Dark Grey	13	1,700	200	1,500	10	3.0	"	21	Blended to obtain quality control
3	10:30	6.1	3.1	Dark Grey	14	1,400	150	1,250	10	3.2	"	21	composite sample for medium dosage
4	11:00	6.1	3.1	Dark Grey	14	1,500	180	1,320	10	3.1		22	
5	11:30	5.9	3.1	Dark Grey	14	1,500	170	1,330	10	3.15	"	21	
6	1:00 pm	5.9	3.1	Dark Grey	14	1,500	150	1,350	10	3.1	"	22	Blended to obtain non-quality control
7	1:30	5.9	3.1	Dark Grey	14	1,550	160	1,390	10	3.1	"	22	composite sample for medium dosage
8	2:00	5.85	3.1	Dark Grey	-	1,700	195	1,505	10	2.9	"	-	
9	2:25	5.85	3.1	Dark Grey	-	2,500	280	2,220	10	2.0	"	-	
10	2:55	5.85	3.1	Dark Grey	17	1,000	120	880	10	3.65	Brown	22	
11	3:25	5.85	3.1	Dark Grey	-	800	60	740	10	4.1	Brown	22	

*From BIF Chart (based on quantity of sludge initially pumped into reaction chamber).
+Sample identification used in Appendix E.

TABLE A3. DATA FOR PURIFAX TESTS ON WILLIMANTIC SLUDGE

Run No.	Time	Raw Sludge pH	Raw Sludge % Solids	Raw Sludge Color	Raw Sludge °C	Chlorine Application (mg/l) Dosage *	Residual	Demand	Purifax Treated Sludge Treat. Period (min)	pH	Color	°C	Identification and Comments+
1	10:30 am	6.2	4.8	Dark Grey	10	4,300	350	3,950	10	3.35	Medium Brown	22	High "A" - Sludge was heavy textured
2	11:00	6.2	5.0	Dark Grey	-	3,000	320	2,680	10	3.6	Grayish Brown	22	High "B"
3	1:05pm	6.3	5.0	Dark Grey	13	2,500	240	2,260	10	3.8	Ash Gray	22	Value malfunctioned
4	1.50	6.3	5.0	Dark Grey	-	1,500	170	1,330	10	4.3	Gray	-	Medium "A"
5	2:15	6.3	5.0	Dark Grey	-	1,000	100	900	10	4.6	Gray	-	Low "A"
6	2:40	6.2	5.0	Dark Grey	-	800	70	730	10	4.7	Gray	-	Low "B"
7	3:05	6.2	5.0	Dark Grey	-	1,600	190	1,410	10	4.5	Gray	-	Medium "B"

* From BIF chart (based on initial quantity of sludge pumped into reaction chamber).
+ Sample identification used in Appendix E.

APPENDIX B

PROCEDURES FOR SAMPLE HANDLING

COLLECTION TECHNIQUES

All sludge samples from the three treatment plants were handled identically in order to provide uniform conditions. Prior to visiting each plant for sludge pickup, the collection personnel talked with the operators and obtained recent operating data for the selected plant. From the discussion with the operators and operating data, a theoretical ratio of primary/waste activated sludge was determined. Also, operating procedures at the plants were thoroughly reviewed to become familiar with the full treatment processes (which may be important in explaining differences in data).

Upon arriving at the individual plants, recent operating conditions were discussed to ensure that there were no unusual conditions that might effect the study. Primary and waste activated sludges were collected separately. The following steps were used to collect all sludge:

(1) Sludge sample lines were allowed to run for at least 5 minutes to clear the line and obtain a fresh sample.

(2) A 75-liter galvanized-steel container was filled with sludge. This ensured a representative sample. A sample of each sludge type was taken for total solids and volatile solids determination.

(3) Using a metal ladle, sludge was transferred from the steel container to 20-liter glass bottles. (This step was done to simplify transportation.)

(4) When the bottle was filled, a sheet of teflon film was placed between the sludge and cap to prevent contamination. The bottle was then immediately placed in an ice chest and packed in ice.

Preparing for Test

Upon arriving in Boston (the same day the sludge was collected), mixing was immediately started. From the individual plant operating records, a ratio of primary/waste activated sludge was determined. Pittsfield sludge was calculated to be 67 percent primary to 33 percent activated sludge by volume. The Amherst and Willimantic sludges were both mixed 60 percent primary to 40 percent activated sludge by volume.

The calculated volume of each sludge type was then poured from the glass

bottles to a large, clean galvanized-steel container. A small mixer was used to mix the sludge for 45 minutes. Intermittently, a large wooden paddle was used to help mix the sludge. After the mixing was completed, the sludge was immediately transferred into 20-liter glass bottles and preserved with ice. (Transfer to smaller containers was necessary in order to place the sludge in ice chests.) A 200-ml sample of the mixture was taken for total solids and volatile solids determination.

The mixed sludge was divided up into three parts for distribution: Purifax, heat treatment, and digestion. Collection, transportation, and mixing took the whole day, so the samples were stored overnight before distribution the next day.

While the sludge mixing was taking place, the digested sludge seed was carefully proportioned out into the five digester bottles. After the digested sludge seed was added to the digesters, the bottles were sealed and purged with nitrogen to remove all the air. Sludge mixing was not started at this time; the digesters were left this way to allow the sludge to warm up and stabilize before adding the raw sludge.

Throughout the collection, handling, mixing, and distribution period, every attempt was made to prevent contamination, especially from plastics. Some plastic containers had to be used, however. In the Amherst series, field blanks using deionized, distilled water were run through the systems to help identify contamination.

Sludge Sampling Procedures

The sludge sampling schedule was broken down into four areas: feed samples; digestion samples; heat-treatment samples; and Purifax samples. All samples were stored in chests with ice or in refrigerators until analysis.

Anaerobic Digestion -- Digestion samples were taken at the end of the digestion period. All digesters with sludge for a given plant were shut down at the same time to assure uniformity.

Heat Treatment -- Due to the small capacity of the pilot unit (0.8 liter) and the large volume requirement of the analyses, several runs were necessary to obtain enough product for testing. The products were composited and mixed.

Purifax -- Upon arriving at Warwick, RI, for testing, the sludge was removed from the ice chest and transferred to a mixing tank. Enough sludge was then homogenized in a blender to produce a run with the Purifax unit. The output of the unit varied from 2.2 to 2.5 liters. The time, chlorine dosage, volumes, and other pertinent information was recorded and noted for later use.

Centrifuging Test Samples

An IEC Model No. 2347 centrifuge was used to produce cake and centrate. The sludge products had to be divided into four 1-liter bottles and centrifuged for periods ranging from 30 to 60 minutes. The volume of centrate was carefully measured and recorded. The four centrates and four cakes were composited and were well mixed prior to analysis.

APPENDIX C

NOTES ON ANALYTICAL METHODS

TESTING LABORATORIES

Four laboratories were used to complete the testing. These were Camp Dresser & McKee (CDM), Boston and Milwaukee; Research Triangle Institute (RTI), Research Triangle Park, North Carolina; and Arthur D. Little, Inc., (ADL), Cambridge, Massachusetts.

ADL and RTI were responsible for specialized analyses on selected samples from the Amherst series. RTI's methods included glass capillary electron impact and negative chemical ionization gas chromatography mass spectrometry. ADL analyzed the samples for organic priority pollutants and prepared extracts for the RTI tests.

CDM's Boston laboratory was responsible for maintenance tests for anaerobic digesters and final product testing. The Milwaukee laboratory prepared and analyzed the leachates and conducted the TOC analysis for all the final product testing.

SOLIDS AND CHEMICAL OXYGEN DEMAND

Sludge, centrate, and cake samples were analyzed for total solids, total volatile solids, total suspended solids, chemical oxygen demand (COD), total organic carbon (TOC), soluble COD, and soluble TOC.

The analyses were performed by procedures specified in Standard Methods for the Examination of Water and Wastewater, 14th Edition APHA/AWWA/WPCF; and/or by the U.S. EPA Manual for Chemical Analysis of Water and Wastes, March, 1979. Parameters defined as soluble were performed on samples filtered through 0.1 μ membrane filters.

ORGANIC AND INORGANIC CHLORINE

The centrate and cake samples were analyzed for organic and inorganic chlorine by the use of various organic combustion techniques and sample filtration methods with subsequent quantification of inorganic chlorine (i.e., chloride) in resultant sample products. The chloride determinations were performed by the micro-coulometric titrimetic technique using a Model No. 4-2500 chloridometer manufactured by Buchler Instruments, Inc.

1. Total Inorganic Chlorine

   a. Cake samples -- A known weight of sample is heated to 100°C with distilled water for several minutes to leach adsorbed inorganic chlorine from the solids. The mixture is filtered through a Whatman No. 40 filter paper. The solids are leached with additional rinses of heated water (minimum of two times) until the filtrate is determined to be chloride free. The filtrate and washings are combined, brought to a known volume, and analyzed for chlorine. Results are reported as percent by weight, dry basis.

   b. Centrate samples -- A known volume of sample is filtered through a Whatman No. 40 filter paper and the filtrate is analyzed for chloride. Results are reported as milligrams per liter.

2. Insoluble Organic Chlorine

   a. Cake samples -- The solids retained on the filter from Step 1.a are vacuum filtered to remove all washings and are air dried for three days. The organic chlorine in the dried sample is converted to chloride by the sodium peroxide bomb ignition method specified as ASTM Designation E256-74 in the 1979 Annual Book of ASTM Standards Part 30, American Society for Testing & Materials. The contents of the bomb after ignition are dissolved in water, neutralized with nitric acid, and brought to a known volume. The resultant aqueous sample is analyzed for chloride. Results are reported as percent by weight, dry basis.

   b. Centrate samples -- The sample residue retained on the filter from Step 1.b is rinsed with heated water to remove any trace amounts of inorganic chlorine, vacuum filtered to remove all washings, and air dried for three days. The organic chlorine in the filtered residue is converted to chloride by combustion of the total filter using the oxygen-flask ignition technique according to the method specified as ASTM Designation E442-74 in the same reference cited in Step 2.a. The resultant aqueous solution (of known volume) is analyzed for chloride. Results are reported as milligrams per liter.

3. Soluble Organic Chlorine

   a. Cake samples -- A known weight of sample is mixed with water and filtered through a Whatman No. 40 filter paper. The solids are rinsed with additional amounts of water (minimum of three times), and the entire filtrate and washings are extracted with a mixture of 85% hexane/15% diethylether three times, on a one part solvent to five parts sample volume basis. The extract is concentrated and totally ignited by the oxygen-flask technique as in Step 2.b. The resultant aqueous solution (of known volume) is analyzed for chloride. Results are reported as percent by weight, dry basis.

   b. Centrate samples -- A known volume of the sample filtrate obtained from Step 1.b is extracted with a mixture of 85% hexane/15% diethy-

lether by the same procedure described in Step 3.a. The extract is also concentrated and analyzed similarly. Results are reported as milligrams per liter.

4. Total Organic Chlorine

Calculated as the sum of the results obtained from the insoluble and soluble organic chlorine determinations.

ORGANIC PRIORITY POLLUTANTS

Arthur D. Little, Inc. (ADL) carried out the analyses for organic priority pollutants on seven sludges and the corresponding cakes from the tests on Amherst sludge: digester seed sludge (from the Ansonia, CT plant), duplicates of feed sludge, a duplicate of medium Purifax (one of which was used for quality control), a heat treatment sample, and a digested sludge sample with 75 percent seed and 25 percent feed. An additional test was conducted on centrate from the Purifax test. The samples were coded as shown on Table C1.

The samples were analyzed for organic priority pollutants in the following categories:

Acidic Extractable Organics (11 species)
Base/Neutral Extractable Organics (49 species)
Pesticides and PCB Organics (25 species)

In addition, the sludge samples were analyzed for Volatile Organics (30 species). The quality control data obtained for this program are presented in Appendix D.

The procedures used to analyze the samples are based on the Federal Register, December 3, 1971[1], the EPA Screening Protocol[2], the EPA-MRI Sludge protocol[3], the EPA Interim Procedures for Sediments and Fish[4], and the EPA 1978 Quality

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1 Federal Register, Part III, Environmental Protection Agency, "Guidelines Establishing Test Procedures for the Analysis of Pollutants; Proposed Regulations", Vol. 44, No. 233, Monday, December 3, 1979.

2 "Sampling and Analysis Procedures for Screening of Industrial Effluents for Priority Pollutants", U.S. EPA, EMSL, Cincinnati, Ohio, March, 1977, Revised April 1977.

3 "Development of Analytical Test Procedures for the Measurement of Organic Priority Pollutants in Sludges and Sediments", Special Report No. 1, "Analytical Protocol for Screening Publicly-Owned Treatment Works (POTW) Sludges for Organic Priority Pollutants", prepared by Midwest Research Institute for EPA, EMSL, Cincinnati, Ohio, June 26, 1979.

4 "Interim Methods for the Sampling and Analysis of Priority Pollutants in Sediments and Fish Tissue", U.S. EPA, EMSL, Cincinnati, Ohio, August 1977.

TABLE C1. SAMPLE CODING FOR ORGANIC SAMPLES*

Sample	Cakes	Sludges	Centrate	Leachates†
Medium Purifax (quality control)	500	506	513	6961
Medium Purifax	501	507		6962
Feed	502	508		6694
Feed	503	509		6695
Digester seed (from Ansonia, CT)	504	510		6693
Heat Treatment	505	511		6960
Digested	515	514		AM-D 8+9

* The blank quality control sample was labeled 512.

† Coding for analyses of leachate conducted by the Research Triangle Institute (RTI).

TABLE C2. OPERATING PARAMETERS FOR ANALYSIS OF EXTRACTS BY PULSED POSITIVE ION NEGATIVE ION CHEMICAL IONIZATION GC/MS/COMP

Parameter	Setting, etc.
1. GC Capillary	50M, wide bore (0.32 mm I.D.), SP2100 fused silica
2. Column Temperature	150°C to 250°C @ 3°C/min
3. Capillary Flow	H_e, 1.0 ml/min
4. Chemical Ionization Reagent Gas	Methane, 0.20-0.22 Torr
5. Electron Multiplier	1400-1500 V

Assurance program[5]. Chemical analysis of the samples included appropriate sample preparation (liquid/liquid extraction, etc.) and subsequent instrumental analysis.

CHLORINATED ORGANICS

The Research Triangle Institute (RTI) conducted analyses using pulsed positive ion negative ion chemical ionization/capillary gas chromatography/mass spectrometry/computer (PPINICHI/GC/MS/COMP). This technique is highly selective and sensitive to chlorine atoms. The analyses were conducted on acid and base extracts of the sludges and cakes that were analyzed for priority pollutants and on leachates from the cakes. See Table C1 for the codes used to identify samples. RTI added the letter A for acid extracts and B for base extracts.

The analytical method provides simultaneous positive ion and negative ion mass spectra on a single injection of extract. From this information, it is possible to selectively detect the compounds that have a propensity to capture electrons (e.g., chlorinated organics). For the analyses indicating the presence of chlorinated material, a data processing system was used to prepare single ion profiles of the m/z 35 and 37 ions to identify the compounds.

Extraction of Leachates

The methods employed for the extraction of leachates were those used for analysis of priority pollutant extractables for wastewater.

Instrumental Analysis

The extracts were analyzed by PPINICHI/GC/MS/COMP techniques. A Finnigan 4023 low resolution quadrupole instrument with a mass range to 1000 and unit resolution was used. A fused silica capillary coated with SP2100 was directly coupled to the ion source. A splitless injection technique was employed. An INCOS 2300 Data System (NOVA 3/12) with a 32K central processor was interfaced with the GC/MS system. Table C2 presents the operating parameters for the analysis of extracts by pulsed positive ion negative ion chemical ionization GC/MS/COMP.

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5 "Procedures for Preliminary Evaluation of Analytical Methods to be Used in the Verification Phase of the Effluent Guidelines Division BAT Review", U.S. EPA, EMSL, Cincinnati, Ohio, March 1978.

APPENDIX D

QUALITY ASSURANCE PROGRAM FOR ORGANIC PRIORITY POLLUTANTS

The procedures used for this study were derived from the program recommended by EMSL/EPA in March, 1978.[6]

Each sample designated a quality control sample was split into three aliquots for each compound category. Two aliquots were spiked in duplicate with a solution containing a known concentration of the appropriate standards for that category. These spiked aliquots were labeled "A" and "B" in ADL's Quality Assurance Program. The third unspiked aliquot was analyzed as the unknown "C".

Associated with these three aliquots was a spiked method reference standard, labeled "D" in ADL's program. This standard is an aliquot of "Milli-Q" water (i.e., distilled and deionized water which has been carbon-filtered to remove organics). The method reference standard was spiked with the standard spiking solution at the same concentration level as "A" and "B".

Method blanks, labeled "F" and consisting of unspiked Milli-Q water, were also extracted, concentrated, and analyzed for each compound category. Extraction, concentration, and analysis procedures followed for the Quality Control samples are detailed in Appendix C.

In order to determine both actual priority pollutant values for the unknowns, as well as recoveries of the spiked samples, calibration standards covering the projected range were prepared and analyzed by GC/MS. From these known values, a calibration curve for each compound was prepared, relating the measured variable (i.e., peak area) to the known concentration. The calibration data was gathered throughout the period of analysis of samples in that category. Thus, daily calibration checks were included in the initial calibration curves.

Priority pollutants analyses have been carried out in the Arthur D. Little, Inc. laboratory for several years. Acceptable recovery ranges have been established, based on many prior studies. Present Quality Control samples are judged in or out of control based on these established individual mean percent recovery and standard deviation results.

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6 "Procedures for Preliminary Evaluation of Analytical Methods to be Used in the Verification Phase of the Effluents Guidelines Division BAT Review", U.S. EPA, EMSL, Cincinnati, Ohio, March 1978.

TABLE D1. QUALITY CONTROL DATA
SPIKED SLUDGE SAMPLES, PERCENT RECOVERY
PURGEABLE (VOLATILES)

SAMPLE NUMBER		506A	506A	506B		Average Recovery	Standard Deviation
101.	Chloromethane	40	0	0		13	23.1
102.	Dichlorodifluoromethane	35	5	2		14	18.2
103.	Bromomethane	110	21	21		51	51.4
104.	Vinyl chloride	55	25	25		35	17.3
105.	Chloroethane	60	35	37		34	27.6
106.	Methylene chloride	5	55	55		38	28.9
107.	Acrolein	76	68	60		68	8.0
108.	Trichlorofluoromethane	12	70	70		51	33.5
109.	Acrylonitrile	62	44	36		47	13.3
110.	1,1–Dichloroethylene	53	70	70		66	6.9
111.	1,1–Dichloroethane	67	77	75		73	5.3
112.	Trans–1,2–dichloroethylene	65	75	70		70	5.0
113.	Chloroform	60	75	70		68	7.6
114.	1,2–Dichloroethane	50	70	70		63	11.5
115.	1,1,1–Trichloroethane	75	85	85		82	5.8
116.	Carbon tetrachloride	79	80	80		80	0.6
117.	Bromodichloromethane	87	95	90		91	4.0
118.	1,2–Dichloropropane	45	65	65		91	4.0
119.	Trans–1,3–dichloropropylene	83	65	70		73	9.3
120.	Trichloroethylene	81	80	80		80	0.6
121.	Benzene	77	85	85		82	4.6
122.	Cis–1,3–dichloropropylene	92	85	90		89	3.6
123.	Dibromochloromethane	135	100	105		113	18.9
124.	1,1,2–Trichloroethane	86	80	80		82	3.5
125.	Bromoform	200	125	130		152	41.9
126.	1,1,2,2–Tetrachloroethane	82	95	90		89	3.6
127.	1,1,2,2–Tetrachloroethylene	81	100	95		92	9.8
128.	Toluene	98	100	100		99	1.2
129.	Chlorobenzene	94	95	95		95	0.6
130.	Ethyl benzene	82	95	95		91	7.5

TABLE D2. QUALITY CONTROL DATA
METHOD REFERENCE STANDARDS, PERCENT RECOVERY
PURGEABLE (VOLATILES)

SAMPLE NUMBER		506D	506D'		Average Recovery		Range	
101.	Chloromethane	21	18		20		3	
102.	Dichlorodifluoromethane	27	0		14		27	
103.	Bromomethane	102	26		64		76	
104.	Vinyl chloride	46	20		33		26	
105.	Chloroethane	64	31		47		33	
106.	Methylene chloride	81	64		72		17	
107.	Acrolein	75	55		65		20	
108.	Trichlorofluoromethane	12	65		49		53	
109.	Acrylonitrile	56	37		46		19	
110.	1,1–Dichloroethylene	53	70		61		17	
111.	1,1–Dichloroethane	65	75		70		10	
112.	Trans–1,2–dichloroethylene	65	75		70		10	
113.	Chloroform	75	85		80		10	
114.	1,2–Dichloroethane	50	70		60		20	
115.	1,1,1–Trichloroethane	70	85		78		15	
116.	Carbon tetrachloride	65	85		75		20	
117.	Bromodichloromethane	85	95		90		10	
118.	1,2–Dichloropropane	50	65		58		15	
119.	Trans–1,3–dichloropropylene	83	85		94		2	
120.	Trichloroethylene	79	80		80		1	
121.	Benzene	74	80		77		6	
122.	Cis–1,3–dichloropropylene	96	90		93		6	
123.	Dibromochloromethane	140	100		120		40	
124.	1,1,2–Trichloroethane	88	80		84		8	
125.	Bromoform	200	125		161		75	
126.	1,1,2,2–Tetrachloroethane	83	90		86		7	
127.	1,1,2,2–Tetrachloroethylene	74	90		82		16	
128.	Toluene	90	100		95		10	
129.	Chlorobenzene	96	95		96		1	
130.	Ethyl benzene	82	95		90		13	

TABLE D3. QUALITY CONTROL DATA
SPIKED SLUDGE SAMPLES, PERCENT RECOVERY
ACIDS

	SAMPLE NUMBER	506A		506B		Average Recovery		Range
201.	2–Chlorophenol	40		56		48		16
202.	2–Nitrophenol	32		48		40		16
203.	Phenol	32		48		40		16
204.	2,4–Dimethylphenol	32		48		40		16
205.	2,4–Dichlorophenol	32		48		40		16
206.	2,4,6–Trichlorophenol	40		40		40		0
207.	4–Chloro–3–cresol	32		32		32		0
208.	2,4–Dinitrophenol	0		0		0		–
209.	4,6–Dinitro–2–cresol	8		8		8		0
210.	Pentachlorophenol	32		48		40		16
211.	4–Nitrophenol	0		0		0		–

TABLE D4. QUALITY CONTROL DATA
METHOD REFERENCE STANDARD, PERCENT RECOVERY
(USING SLUDGE ANALYTICAL TECHNIQUES)
ACIDS

	SAMPLE NUMBER	506D							
201.	2–Chlorophenol	32							
202.	2–Nitrophenol	32							
203.	Phenol	32							
204.	2,4–Dimethylphenol	40							
205.	2,4–Dichlorophenol	40							
206.	2,4,6–Trichlorophenol	56							
207.	4–Chloro–3–cresol	40							
208.	2,4–Dinitrophenol	24							
209.	4,6–Dinitro–2–cresol	24							
210.	Pentachlorophenol	56							
211.	4–Nitrophenol	104							

TABLE D5. QUALITY CONTROL DATA
SPIKED CAKE SAMPLES, PERCENT RECOVERY
ACIDS

SAMPLE NUMBER	500A		500B		Average Recovery		Range
201. 2–Chlorophenol	44		52		48		8
202. 2–Nitrophenol	46		50		48		4
203. Phenol	48		58		53		10
204. 2,4–Dimethylphenol	54		62		58		8
205. 2,4–Dichlorophenol	56		60		58		4
206. 2,4,6–Trichlorophenol	66		60		63		6
207. 4–Chloro–3–cresol	48		56		52		8
208. 2,4–Dinitrophenol	100		62		81		38
209. 4,6–Dinitro–2–cresol	52		36		44		16
210. Pentachlorophenol	68		44		56		24
211. 4–Nitrophenol	18		0		9		18

*Percent Recovery

TABLE D6. QUALITY CONTROL DATA
METHOD REFERENCE STANDARD, PERCENT RECOVERY
(USING CAKE ANALYTICAL TECHNIQUES)
ACIDS

SAMPLE NUMBER		500D						
201. 2–Chlorophenol		48						
202. 2–Nitrophenol		74						
203. Phenol		42						
204. 2,4–Dimethylphenol		2						
205. 2,4–Dichlorophenol		36						
206. 2,4,6–Trichlorophenol		52						
207. 4–Chloro–3–cresol		22						
208. 2,4–Dinitrophenol		166						
209. 4,6–Dinitro–2–cresol		64						
210. Pentachlorophenol		54						
211. 4–Nitrophenol		94						

TABLE D7. QUALITY CONTROL DATA
SPIKED SLUDGE SAMPLES, PERCENT RECOVERY
BASE/NEUTRALS

SAMPLE NUMBER		506A		506B		Average Recovery		Range
301.	1,3 Dichlorobenzene	41		31		36		10
302.	1,4 Dichlorobenzene							
303.	1,2 Dichlorobenzene							
304.	Hexachloroethane	38		37		38		1
305.	Bis(chloromethyl)ether					-		-
306.	Bis(2-chloroethyl) ether	48		37		54		11
307.	Bis(2-chloroisopropyl) ether	NOT ADDED						
308.	N-Nitrosodimethylamine	49		29		39		20
309.	Nitrosodi-n-propylamine	9		.3		4		7.7
310.	Nitrobenzene	30		17		36		13
311.	Hexachlorobutadiene	44		35		39		9
312.	1,2,4-Trichlorobenzene	46		34		40		12
313.	2-Chloroethyl vinyl ether					-		-
314.	Bis(2-chloroethoxy) methane	32		16		24		16
315.	Naphthalene	34		22		26		12
316.	Isophorone	22		16		18		6
317.	Hexachlorocyclopentadiene					-		-
318.	2-Chloronaphthalene	38		23		30		15
319.	Acenaphthylene	15		.3		8		14.7
320.	Acenaphthene	37		24		30		13
321.	Dimethyl phthalate	47		26		33		14
322.	2,6-Dinitrotoluene	84		57		70		27
323.	4-Chlorophenyl phenyl ether	46		36		41		10
324.	Fluorene	41		29		35		12
325.	2,4-Dinitrotoluene	56		40		48		16
326.	Diethyl phthalate	44		28		36		16
327.	1,2-Diphenylhydrazine	40		32		36		8
328.	N-Nitrosodiphenylamine	49		40		44		9
329.	Hexachlorobenzene	42		32		37		10
330.	4-Bromophenyl phenyl ether	49		38		34		11

TABLE D7. QUALITY CONTROL DATA
SPIKED SLUDGE SAMPLES, PERCENT RECOVERY
BASE/NEUTRALS
(continued)

SAMPLE NUMBER		506A		506B		Average Recovery		Range
331.	Anthracene	72		54		63		18
332.	Phenanthrene							
333.	Di-n-butyl phthalate	26		15		20		11
334.	Fluoranthene	30		21		25		9
335.	Pyrene	27		20		24		7
336.	Benzidine					-		-
337.	Butyl benzyl phthalate	4		3		4		1
338.	Bis(2-ethylhexyl) phthalate	72		16		54		56
339.	Di-n-octyl phthalate							
340.	Chrysene	36		24		30		12
341.	Benzo(a)anthracene							
342.	3,3'-Dichlorobenzidine					-		-
343.	Benzo(b)fluoranthene	94		80		87		14
344.	Benzo(k)fluoranthene							
345.	Benzo(a)pyrene	53		47		50		6
346.	Indeno (1,2,3-c,d) pyrene	77		73		75		4
347.	Dibenzo (a,h) Anthracene	66		60		63		6
348.	Benzo (g,h,i) perylene	72		64		68		8

TABLE D8. QUALITY CONTROL DATA

METHOD REFERENCE STANDARD, PERCENT RECOVERY
(USING SLUDGE ANALYTICAL TECHNIQUES)

BASE/NEUTRALS

	SAMPLE NUMBER			506D					
301.	1,3 Dichlorobenzene			33					
302.	1,4 Dichlorobenzene								
303.	1,2 Dichlorobenzene								
304.	Hexachloroethane			42					
305.	Bis(chloromethyl)ether								
306.	Bis(2–chloroethyl) ether			45					
307.	Bis(2–chloroisopropyl) ether	NOT ADDED							
308.	N–Nitrosodimethylamine			0					
309.	Nitrosodi-n-propylamine			0					
310.	Nitrobenzene			20					
311.	Hexachlorobutadiene			37					
312.	1,2,4–Trichlorobenzene			27					
313.	2–Chloroethyl vinyl ether			–					
314.	Bis(2–chloroethoxy) methane			15					
315.	Naphthalene			24					
316.	Isophorone			13					
317.	Hexachlorocyclopentadiene			–					
318.	2–Chloronaphthalene			29					
319.	Acenaphthylene			9					
320.	Acenaphthene			34					
321.	Dimethyl phthalate			1					
322.	2,6–Dinitrotoluene			57					
323.	4–Chlorophenyl phenyl ether			61					
324.	Fluorene			48					
325.	2,4–Dinitrotoluene			62					
326.	Diethyl phthalate			8					
327.	1,2–Diphenylhydrazine			40					
328.	N–Nitrosodiphenylamine			49					
329.	Hexachlorobenzene			59					
330.	4–Bromophenyl phenyl ether			58					

TABLE D8. QUALITY CONTROL DATA

METHOD REFERENCE STANDARD, PERCENT RECOVERY
(USING SLUDGE ANALYTICAL TECHNIQUES)

BASE/NEUTRALS

(continued)

SAMPLE NUMBER			506D					
331. Anthracene / 332. Phenanthrene			105					
333. Di-n-butyl phthalate			19					
334. Fluoranthene			66					
335. Pyrene			69					
336. Benzidine			-					
337. Butyl benzyl phthalate			6					
338. Bis(2-ethylhexyl) phthalate / 339. Di-n-octyl phthalate			50					
340. Chrysene / 341. Benzo(a)anthracene			108					
342. 3,3'-Dichlorobenzidine			53					
343. Benzo(b)fluoranthene / 344. Benzo(k)fluoranthene			128					
345. Benzo(a)pyrene			70					
346. Indeno (1,2,3-c,d) pyrene			104					
347. Dibenzo (a,h) Anthracene			98					
348. Benzo (g,h,i) perylene			88					

TABLE D9. QUALITY CONTROL DATA
SPIKED CAKE SAMPLES, PERCENT RECOVERY
BASE/NEUTRALS

SAMPLE NUMBER	500A		500B		Average Recovery		Range
301. 1,3 Dichlorobenzene 302. 1,4 Dichlorobenzene 303. 1,2 Dichlorobenzene	48		40		44		8
304. Hexachloroethane	26		28		27		2
305. Bis(chloromethyl)ether					-		-
306. Bis(2-chloroethyl) ether	79		82		80		3
307. Bis(2-chloroisopropyl) ether	Not Added						
308. N-Nitrosodimethylamine	6		67		36		61
309. Nitrosodi-n-propylamine	48		72		60		24
310. Nitrobenzene	76		83		80		7
311. Hexachlorobutadiene	38		32		35		6
312. 1,2,4-Trichlorobenzene	66		60		63		6
313. 2-Chloroethyl vinyl ether					-		-
314. Bis(2-chloroethoxy) methane	99		99		99		0
315. Naphthalene	67		71		69		4
316. Isophorone	45		46		46		1
317. Hexachlorocyclopentadiene					-		-
318. 2-Chloronaphthalene	76		71		74		5
319. Acenaphthylene	73		71		72		2
320. Acenaphthene	71		69		70		2
321. Dimethyl phthalate	78		76		77		2
322. 2,6-Dinitrotoluene	116		114		115		2
323. 4-Chlorophenyl phenyl ether	54		41		48		13
324. Fluorene	67		66		66		1
325. 2,4-Dinitrotoluene	104		102		103		2
326. Diethyl phthalate	138		124		131		14
327. 1,2-Diphenylhydrazine	72		70		71		2
328. N-Nitrosodiphenylamine	55		62		58		7
329. Hexachlorobenzene	48		40		44		8
330. 4-Bromophenyl phenyl ether	50		47		48		3

TABLE D9. QUALITY CONTROL DATA
SPIKED CAKE SAMPLES, PERCENT RECOVERY
BASE/NEUTRALS
(continued)

SAMPLE NUMBER	500A		500B		Average Recovery		Range
331. Anthracene / 332. Phenanthrene	101		99		100		2
333. Di-n-butyl phthalate	56		40		48		16
334. Fluoranthene	42		39		40		3
335. Pyrene	42		49		40		3
336. Benzidine					-		-
337. Butyl benzyl phthalate	12		10		11		2
338. Bis(2-ethylhexyl) phthalate / 339. Di-n-octyl phthalate	34		18		26		16
340. Chrysene / 341. Benzo(a)anthracene	58		49		53		9
342. 3,3'-Dichlorobenzidine			.		-		-
343. Benzo(b)fluoranthene / 344. Benzo(k)fluoranthene	65		60		62		5
345. Benzo(a)pyrene	29		28		28		1
346. Indeno (1,2,3-c,d) pyrene	38		35		36		3
347. Dibenzo (a,h) Anthracene	36		34		35		2
348. Benzo (g,h,i) perylene	28		32		35		6

TABLE D10. QUALITY CONTROL DATA

METHOD REFERENCE STANDARD, PERCENT RECOVERY
(USING CAKE ANALYTICAL TECHNIQUES)

BASE/NEUTRALS

	SAMPLE NUMBER		500D						
301.	1,3 Dichlorobenzene		18						
302.	1,4 Dichlorobenzene								
303.	1,2 Dichlorobenzene								
304.	Hexachloroethane		17						
305.	Bis(chloromethyl)ether		-						
306.	Bis(2-chloroethyl) ether		36						
307.	Bis(2-chloroisopropyl) ether		Not Added						
308.	N-Nitrosodimethylamine		1						
309.	Nitrosodi-n-propylamine		40						
310.	Nitrobenzene		44						
311.	Hexachlorobutadiene		23						
312.	1,2,4-Trichlorobenzene		31						
313.	2-Chloroethyl vinyl ether		-						
314.	Bis(2-chloroethoxy) methane		56						
315.	Naphthalene		41						
316.	Isophorone		23						
317.	Hexachlorocyclopentadiene		-						
318.	2-Chloronaphthalene		57						
319.	Acenaphthylene		53						
320.	Acenaphthene		61						
321.	Dimethyl phthalate		70						
322.	2,6-Dinitrotoluene		86						
323.	4-Chlorophenyl phenyl ether		73						
324.	Fluorene		74						
325.	2,4-Dinitrotoluene		86						
326.	Diethyl phthalate		84						
327.	1,2-Diphenylhydrazine		84						
328.	N-Nitrosodiphenylamine		46						
329.	Hexachlorobenzene		70						
330.	4-Bromophenyl phenyl ether		73						

TABLE D10. QUALITY CONTROL DATA

METHOD REFERENCE STANDARD, PERCENT RECOVERY
(USING CAKE ANALYTICAL TECHNIQUES)

BASE/NEUTRALS

(continued)

SAMPLE NUMBER		500D						
331. Anthracene		156						
332. Phenanthrene								
333. Di-n-butyl phthalate		84						
334. Fluoranthene		82						
335. Pyrene		82						
336. Benzidine		-						
337. Butyl benzyl phthalate		28						
338. Bis(2-ethylhexyl) phthalate		88						
339. Di-n-octyl phthalate								
340. Chrysene		142						
341. Benzo(a)anthracene								
342. 3,3'-Dichlorobenzidine		30						
343. Benzo(b)fluoranthene		150						
344. Benzo(k)fluoranthene								
345. Benzo(a)pyrene		70						
346. Indeno (1,2,3-c,d) pyrene		84						
347. Dibenzo (a,h) Anthracene		86						
348. Benzo (g,h,i) perylene		84						

TABLE D11. QUALITY CONTROL DATA
SPIKED SLUDGE SAMPLES, PERCENT RECOVERY
PESTICIDES

		506A	506B	Average Recovery	Range
401.	alpha-BHC	113	121	117	8
402.	gamma-BHC	87	35	61	52
403.	Heptachlor	174*	176*	175	2
404.	beta-BHC	268*	268*	268	0
405.	delta-BHC	122	134	128	12
406.	Aldrin	96	90	93	6
407.	Heptachlor epoxide	94	94	94	0
408.	Endosulfan I.	89	112	100	23
409.	DDE	84	84	84	0
410.	Dieldrin	71	65	68	6
411.	Endrin	59	59	59	0
412.	DDD	101	91	96	10
413.	Endosulfan II				
414.	DDT	66	76	71	10
415.	Endrin aldehyde	33	41	37	8
416.	Endosulfan sulfate	58	62	60	4
417.	Chlordane				
418.	Toxaphene				
419.	PCB-1221				
420.	PCB-1232				
421.	PCB-1242				
422.	PCB-1248				
423.	PCB-1254	120	120	120	0
424.	PCB-1260				
425.	PCB-1016				

*Suspected Contamination

TABLE D12. QUALITY CONTROL DATA

METHOD REFERENCE STANDARD, PERCENT RECOVERY
(USING SLUDGE ANALYTICAL TECHNIQUES)

PESTICIDES

				506D					
401.	alpha-BHC			67					
402.	gamma-BHC			61					
403.	Heptachlor			53					
404.	beta-BHC			74					
405.	delta-BHC			96					
406.	Aldrin			46					
407.	Heptachlor epoxide			54					
408.	Endosulfan I.			95					
409.	DDE			44					
410.	Dieldrin			17					
411.	Endrin			33					
412.	DDD			138					
413.	Endosulfan II								
414.	DDT			34					
415.	Endrin aldehyde								
416.	Endosulfan sulfate								
417.	Chlordane								
418.	Toxaphene								
419.	PCB–1221								
420.	PCB–1232								
421.	PCB–1242								
422.	PCB–1248								
423.	PCB–1254			20					
424.	PCB–1260								
425.	PCB–1016								

TABLE D13. QUALITY CONTROL DATA
SPIKED CAKE SAMPLES, PERCENT RECOVERY
PESTICIDES

		500A	500B	Average Recovery	Range
401.	alpha-BHC	92	85	88	7
402.	gamma-BHC	107	95	101	12
403.	Heptachlor	86	81	83	5
404.	beta-BHC	101	90	95	11
405.	delta-BHC	108	102	105	6
406.	Aldrin	83	80	82	3
407.	Heptachlor epoxide	82	76	79	6
408.	Endosulfan I.	83	80	82	3
409.	DDE	88	80	84	8
410.	Dieldrin	75	76	76	1
411.	Endrin	55	54	54	1
412.	DDD	101	93	97	8
413.	Endosulfan II				
414.	DDT	85	83	84	2
415.	Endrin aldehyde	8	40	24	32
416.	Endosulfan sulfate	15	90	52	75
417.	Chlordane				
418.	Toxaphene				
419.	PCB-1221				
420.	PCB-1232				
421.	PCB-1242				
422.	PCB-1248				
423.	PCB-1254	80	80	80	0
424.	PCB-1260				
425.	PCB-1016				

TABLE D14. QUALITY CONTROL DATA

METHOD REFERENCE STANDARD, PERCENT RECOVERY
(USING CAKE ANALYTICAL TECHNIQUES)

PESTICIDES

				500D					
401.	alpha-BHC			86					
402.	gamma-BHC			83					
403.	Heptachlor			80					
404.	beta-BHC			93					
405.	delta-BHC			93					
406.	Aldrin			81					
407.	Heptachlor epoxide			82					
408.	Endosulfan I.			73					
409.	DDE			90					
410.	Dieldrin			56					
411.	Endrin			57					
412.	DDD			48					
413.	Endosulfan II								
414.	DDT			90					
415.	Endrin aldehyde								
416.	Endosulfan sulfate								
417.	Chlordane								
418.	Toxaphene								
419.	PCB—1221								
420.	PCB—1232								
421.	PCB—1242								
422.	PCB—1248								
423.	PCB—1254								
424.	PCB—1260								
425.	PCB—1016								

The sample coding for sludge and cake samples is given in Table D9.

The percent recovery data for both sludge and cake spikes, as well as method reference standards are presented in Tables D1 to D14.

The following are the concentration levels to which the priority pollutants were added to the sludge and cake samples:

	Sludge	Cake
Volatiles	20 µg/l	
Acids	125 µg/l	5000 µg/kg
Base/Neutrals	125 µg/l	5000 µg/kg
Pesticides	100 µg/l	1000 µg/kg
PCB 1254	900 µg/l	9000 µg/kg

The method reference standards (D) were prepared for analysis in the same way that the sludge and cake samples were prepared; therefore, the cake method reference standard (500D) was subjected to sonication and the sludge method reference standard (506D) was subjected to homogenization for acid, base/neutral, and pesticide and PCB analyses.

A listing of terms and calculations of data is given below:

A = field sample, spiked
B = field sample, spiked as replicate of A
C = unknown = field sample, unspiked
D = method reference standards
  = Milli-Q water, spiked at same level as A and B.
F = method blank
  = Milli-Q water, unspiked

Calculations for spiked sludge and cake samples:

$$\text{Percent Recovery} = \left[\frac{\left[\text{Concentration of Spiked Sludge or Cake (A or B)}\right] - \left[\text{Concentration of Sludge or Cake (C)}\right]}{\text{True Spiked Concentration}} \right] \times 100$$

$$\text{Standard deviation of percentages} = \sqrt{\frac{\sum_{i=1}^{n} (P_i - \bar{P})^2}{n - 1}}$$

Range of Percentages = Percent Recovery of A - Percent Recovery of B

Calculations for Method Reference Standards:

$$\text{Percent} = \frac{\text{Concentration of Method Reference Standard (D)}}{\text{True Concentration of Spike}} \times 100$$

The average recoveries calculated for each of the categories of organic priority pollutants from sludge and cake samples follow:

	Sludge	Cake
Volatiles	72%	---
Acids	36%	52%
Base/Neutrals	38%	64%
Pesticides and PCBs	85%	73%

These recoveries are consistent with data obtained from other laboratories with similar samples. Organic priority pollutants that were not detected in the spiked samples are:

305	Bis(chloromethyl) ether
313	2-Chloroethyl vinyl ether
317	Hexachlorocyclopentadiene
336	Benzidine
342	3,3'-Dichlorobenzidiene

Bis(chloromethyl) ether has a very short half-life in water, and therefore is not detected in matrices containing water. Two of the compounds (317, hexachlorocyclopentadiene and 336, benzidiene), not detected; chromatographed poorly (excessive GC peak tailings, as well as extracting poorly) using the EPA methods. 3, 3'-Dichlorobenzidine (342) was not extracted from the sludge and cake samples; however, it was extracted from the two method reference standards. Apparently, for this compound, problems were associated with the sample matrix. It is unknown why 2-chloroethyl vinyl ether was not detected.

If these four compounds (313, 317, 336, and 342) had been present in the sludge and cake samples, they probably would not have been detected unless they were present at concentration levels exceeding 10,000 ug/kg (for cake samples) and 1,000 ug/L (for sludge samples).

The results of analyzing field blanks and method blanks are presented in Tables D15 through D18. Reporting limits for each of the compounds are also given in these tables. No significant levels of priority pollutants were found in the blank samples.

The reporting limits for each type of sample matrix are given in Tables D19 through D22.

TABLE D15. QUALITY CONTROL DATA -
BLANK SAMPLES

PRIORITY POLLUTANT CHEMICAL ANALYSIS (µg/l)
PURGEABLE (VOLATILES)

		Field Blank 512		Method Blank 506F			Reporting Limit	
101.	Chloromethane		–		–		1	
102.	Dichlorodifluoromethane		–		–		1	
103.	Bromomethane		–		–		1	
104.	Vinyl chloride		–		–		1	
105.	Chloroethane		–		–		2	
106.	Methylene chloride		–		3		2	
107.	Acrolein		–		–		10	
108.	Trichlorofluoromethane		–		–		1	
109.	Acrylonitrile		–		–		10	
110.	1,1–Dichloroethylene		–		–		1	
111.	1,1–Dichloroethane		–		–		1	
112.	Trans–1,2–dichloroethylene		–		–		1	
113.	Chloroform		1		–		1	
114.	1,2–Dichloroethane		–		–		1	
115.	1,1,1–Trichloroethane		–		–		1	
116.	Carbon tetrachloride		–		–		1	
117.	Bromodichloromethane		–		–		1	
118.	1,2–Dichloropropane		–		–		1	
119.	Trans–1,3–dichloropropylene		–		–		1	
120.	Trichloroethylene		–		–		1	
121.	Benzene		–		–		1	
122.	Cis–1,3–dichloropropylene		–		–		1	
123.	Dibromochloromethane		–		–		1	
124.	1,1,2–Trichloroethane		–		–		1	
125.	Bromoform		–		–		1	
126.	1,1,2,2–Tetrachloroethane		–		–		1	
127.	1,1,2,2–Tetrachloroethylene		–		–		1	
128.	Toluene		–		–		1	
129.	Chlorobenzene		–		–		1	
130.	Ethyl benzene		–		–		1	

TABLE D16. QUALITY CONTROL DATA - BLANK SAMPLES
PRIORITY POLLUTANT CHEMICAL ANALYSIS (µg/l)
ACIDS

		Field Blank 512				Method Blank 500F			Reporting Limit
201.	2–Chlorophenol		–			–			1
202.	2–Nitrophenol		–			–			1
203.	Phenol		–			–			1
204.	2,4–Dimethylphenol		–			–			1
205.	2,4–Dichlorophenol		–			–			1
206.	2,4,6–Trichlorophenol		–			–			1
207.	4–Chloro–3–cresol		–			–			1
208.	2,4–Dinitrophenol		–			–			1
209.	4,6–Dinitro–2–cresol		–			–			1
210.	Pentachlorophenol		–			–			1
211.	4–Nitrophenol		–			–			1

TABLE D17. QUALITY CONTROL DATA - BLANK SAMPLES
PRIORITY POLLUTANT CHEMICAL ANALYSIS (µg/l)
BASE/NEUTRALS

			Field Blank 513			Method Blank 500F			Reporting Limit
301.	1,3 Dichlorobenzene		-			-			10
302.	1,4 Dichlorobenzene								
303.	1,2 Dichlorobenzene								
304.	Hexachloroethane		-			-			20
305.	Bis(chloromethyl)ether		-			-			-
306.	Bis(2-chloroethyl) ether		-			-			20
307.	Bis(2-chloroisopropyl) ether		-			-			20
308.	N-Nitrosodimethylamine		-			-			10
309.	Nitrosodi-n-propylamine		-			-			10
310.	Nitrobenzene		-			-			10
311.	Hexachlorobutadiene		-			-			20
312.	1,2,4-Trichlorobenzene		-			-			10
313.	2-Chloroethyl vinyl ether		-			-			10
314.	Bis(2-chloroethoxy) methane		-			-			10
315.	Naphthalene		P			P			10
316.	Isophorone		-			-			10
317.	Hexachlorocyclopentadiene		-			-			10
318.	2-Chloronaphthalene		-			-			10
319.	Acenaphthylene		-			-			10
320.	Acenaphthene		-			-			10
321.	Dimethyl phthalate		-			P			10
322.	2,6-Dinitrotoluene		-			-			10
323.	4-Chlorophenyl phenyl ether		-			-			10
324.	Fluorene		-			-			10
325.	2,4-Dinitrotoluene		-			-			10
326.	Diethyl phthalate		-			-			10
327.	1,2-Diphenylhydrazine		-			-			10
328.	N-Nitrosodiphenylamine		-			-			10
329.	Hexachlorobenzene		-			-			10
330.	4-Bromophenyl phenyl ether		-			-			10

TABLE D17. QUALITY CONTROL DATA - BLANK SAMPLES
PRIORITY POLLUTANT CHEMICAL ANALYSIS (μg/1)
BASE/NEUTRALS
(continued)

		Field Blank 513			Method Blank 500F			Reporting Limit
331.	Anthracene	P			P			10
332.	Phenanthrene							
333.	Di-n-butyl phthalate	-			P			10
334.	Fluoranthene	-			P			10
335.	Pyrene	-			-			10
336.	Benzidine	-			-			10
337.	Butyl benzyl phthalate	P			P			10
338.	Bis(2-ethylhexyl) phthalate	24			P			10
339.	Di-n-octyl phthalate							
340.	Chrysene	-			P			10
341.	Benzo(a)anthracene							
342.	3,3'-Dichlorobenzidine	-			-			10
343.	Benzo(b)fluoranthene	-			-			10
344.	Benzo(k)fluoranthene							
345.	Benzo(a)pyrene	-			-			10
346.	Indeno (1,2,3-c,d) pyrene	-			-			20
347.	Dibenzo (a,h) Anthracene	-			-			20
348.	Benzo (g,h,i) perylene	-			-			20
349.	TCDD	-			-			20

P - Present but below reporting limit.

TABLE D18. QUALITY CONTROL DATA - BLANK SAMPLES
PRIORITY POLLUTANT CHEMICAL ANALYSIS (µg/l)
PESTICIDES

		Field Blank 512			Method Blank 500F			Reporting Limit
401.	alpha-BHC		–			–			2
402.	gamma-BHC		–			–			2
403.	Heptachlor		–			–			2
404.	beta-BHC		–			–			2
405.	delta-BHC		–			–			2
406.	Aldrin		–			–			2
407.	Heptachlor epoxide		–			–			2
408.	Endosulfan I.		–			–			2
409.	DDE		–			–			2
410.	Dieldrin		–			–			2
411.	Endrin		–			–			2
412.	DDD		–			–			2
413.	Endosulfan II		–			–			2
414.	DDT		–			–			2
415.	Endrin aldehyde		–			–			2
416.	Endosulfan sulfate		–			–			2
417.	Chlordane		–			–			2
418.	Toxaphene		–			–			2
419.	PCB–1221		–			–			10
420.	PCB–1232		–			–			10
421.	PCB–1242		–			–			10
422.	PCB–1248		–			–			10
423.	PCB–1254		–			–			10
424.	PCB–1260		–			–			10
425.	PCB–1016		–			–			10

TABLE D19. REPORTING LIMITS
PURGEABLE (VOLATILES)

	Compound	Sludge Samples (µg/l)
101.	Chloromethane	1
102.	Dichlorodifluoromethane	1
103.	Bromomethane	1
104.	Vinyl Chloride	1
105.	Chloroethane	2
106.	Methylene Chloride	2
107.	Acrolein	10
108.	Trichlorofluoromethane	1
109.	Acrylonitrile	10
110.	1,1-Dichloroethylene	1
111.	1,1-Dichloroethane	1
112.	Trans-1,2-Dichloroethylene	1
113.	Chloroform	1
114.	1,2-Dichloroethane	1
115.	1,1,1-Trichloroethane	1
116.	Carbon Tetrachloride	1
117.	Bromodichloromethane	1
118.	1,2-Dichloropropane	1
119.	Trans-1,3-Dichloropropylene	1
120.	Trichloroethylene	1
121.	Benzene	1
122.	Cis-1,3-Dichloropropylene	1
123.	Dibromochloromethane	1
124.	1,1,2-Trichloroethane	1
125.	Bromoform	1
126.	1,1,2,2-Tetrachloroethane	1
127.	1,1,2,2-Tetrachloroethylene	1
128.	Toluene	1
129.	Chlorobenzene	1
130.	Ethyl Benzene	1

TABLE D20. REPORTING LIMITS

ACIDS

	Compound	Cake Samples (µg/kg)	Sludge Samples (ug/l)	Liquid Samples (µg/l)
201	2-Chlorophenol	100	10	1
202.	2-Nitrophenol	100	10	1
203.	Phenol	100	10	1
204.	2,4-Dimethylphenol	100	10	1
205.	2,4-Dichlorophenol	100	10	1
206.	2,4,6-Trichlorophenol	100	10	1
207.	4-Chloro-3-Cresol	100	10	1
208.	2,4-Dinitrophenol	100	10	1
209.	4,6-Dinitro-2-Cresol	100	10	1
210.	Pentachlorophenol	100	10	1
211.	4-Nitrophenol	100	10	1

TABLE D21. REPORTING LIMITS

BASE/NEUTRALS

	Compound	Cake Samples (µg/kg)	Sludge Samples (µg/l)	Liquid Samples (µg/l)
301.	1,3-Dichlorobenzene			
302.	1,4-Dichlorobenzene	50	5	10
303.	1,2-Dichlorobenzene			
304.	Hexachloroethane	200	20	20
305.	Bis(chloromethyl)ether	-	-	-
306.	Bis(2-chloroethyl)ether	200	20	20
307.	Bis(2-chloroisopropyl)ether	10,000	1,000	20
308.	n-Nitrosodimethylamine	100	10	10
309.	Nitrosodi-n-propylamine	100	10	10
310.	Nitrobenzene	100	10	10
311.	Hexachlorobutadiene	150	15	20
312.	1,2,4-Trichlorobenzene	100	10	10
313.	2-Chloroethyl Vinyl Ether	10,000	1,000	10
314.	Bis(2-chloroethoxy)methane	100	10	10
315.	Naphthalene	100	10	10
316.	Isophorone	50	5	10
317.	Hexachlorocyclopentadiene	10,000	1,000	10
318.	2-Chloronaphthalene	100	10	10
319.	Acenaphthylene	100	10	10
320.	Acenaphthene	100	10	10
321.	Dimethyl Phthalate	100	10	10
322.	2,6-Dinitrotoluene	100	10	10
323.	4-Chlorophenyl Phenyl Ether	105	15	10
324.	Fluorene	100	10	10
325.	2,4-Dinitrotoluene	150	15	10
326.	Diethyl Phthalate	100	10	10
327.	1,2-Diphenylhydrazine	100	10	10

TABLE D21. REPORTING LIMITS
BASE/NEUTRALS
(continued)

	Compound	Cake Samples (µg/kg)	Sludge Samples (µg/l)	Liquid Samples (µg/l)
328.	n-Nitrosodiphenylamine	150	15	10
329.	Hexachlorobenzene	50	5	10
330.	4-Bromophenyl Phenyl Ether	150	15	10
331.	Anthracene	100	10	10
332.	Phenanthrene			
333.	Di-n-butyl Phthalate	100	10	10
334.	Fluoranthene	200	20	10
335.	Pyrene	200	20	10
336.	Benzidine	10,000	1,000	10
337.	Butyl Benzyl Phthalate	160	20	10
338.	Bis(2-ethylhexyl)phthalate	400	50	10
339.	Di-n-octyl Phthalate			
340.	Chrysene	100	10	10
341.	Benzo(a)anthracene			
342.	3,3'-Dichlorobenzidine	10,000	1,000	10
343.	Benzo(b)fluoranthene	50	10	10
344.	Benzo(k)fluoranthene			
345.	Benzo(a)pyrene	400	50	10
346.	Indeno(1,2,3-c,d)pyrene	700	80	20
347.	Dibenzo(a,h)anthracene	600	60	20
348.	Benzo(g,h,i)perylene	600	70	20
349.	TCDD	600	60	20

TABLE D22. REPORTING LIMITS
PESTICIDES AND PCBs

	Compound	Cake Samples (µg/kg)	Sludge Samples (µg/l)	Liquid Samples (µg/l)
401.	alpha-BHC	25	5	2
402.	gamma-BHC	25	5	2
403.	Heptachlor	25	5	2
404,	beta-BHC	25	5	2
405.	delta-BHC	25	5	2
406.	Aldrin	25	5	2
407.	Heptachlor Epoxide	25	5	2
408.	Endosulfan I	25	5	2
409.	DDE	25	5	2
410.	Dieldrin	25	5	2
411.	Endrin	25	5	2
412.	DDD	25	5	2
413.	Endosulfan II	25	5	2
414.	DDT	25	5	2
415.	Endrin Aldehyde	25	5	2
416.	Endosulfan Sulfate	25	5	2
417.	Chlordane	25	10	10
418.	Toxaphene	25	10	10
419.	PCB-1221	25	10	10
420.	PCB-1232	25	10	10
421.	PCB-1242	25	10	10
422.	PCB-1248	25	10	10
423.	PCB-1254	25	10	10
424.	PCB-1260	25	10	10
425.	PCB-1016	25	10	10

APPENDIX E

RESULTS OF CHEMICAL ANALYSES

The tables in this appendix present results of chemical analyses conducted in this investigation. Appendix C describes the analytical techniques used, and Appendix D presents the quality assurance program for organic priority pollutants. Interpretation of the data is done in the body of the report.

ORGANIC AND INORGANIC CHLORINE

Data on analyses for organic and inorganic chlorine are presented in Tables E1 through E6. Tables E1 and E2 present the analytical results for the sludge feeds to the processes and for the products. Table E1 presents the data for centrifuge cake, and Table E2, for centrate.

Tables E3 through E5, which are based on Tables E1 and E2, present the distribution of organic chlorine between cake and centrate in sludge feeds and products. Table E3 provides the data for Amherst; Table E4, for Pittsfield; and Table E5, for Willimantic.

Table E6 provides the data developed to assess the recovery of total chlorine.

ORGANIC PRIORITY POLLUTANTS

The analytical results for the volatile, acid, base/neutral, and pesticide and PCB priority pollutants are presented in Tables E7 through E11. Each table presents the data for a particular sludge and its corresponding cake or liquid sample. Data are given only for those priority pollutants that were detected in at least one sample. A list of those priority pollutants that were never detected in any sample is given in Table E12.

The analytical results for volatile, acid, and base/neutral priority pollutants are based on gas chromatography/mass spectroscopy analysis. The analytical results for pesticide and PCB priority pollutants are based on gas chromatography/electron capture detection analysis. The results of analyzing quality control samples indicate that these data are valid.

Limitations on Pesticide and PCB Analysis of Sludges and Cakes

There are seven manufactured PCBs as well as 18 pesticides that are categorized as priority pollutants. A single PCB is actually a complicated mix-

TABLE E1. COMPARISON OF CENTRIFUGE SLUDGE CAKES FROM DIFFERENT WASTEWATER PLANTS

SLUDGE SOURCE*		TOTAL SOLIDS (%)			INSOLUBLE ORGANIC CHLORINE (% by wt. dry basis)			SOLUBLE ORGANIC CHLORINE (% by wt. dry basis)			TOTAL ORGANIC CHLORINE (% by wt. dry basis)			TOTAL INORGANIC CHLORINE (% by wt. dry basis)			CHEMICAL OXYGEN DEMAND (COD) (% by wt. dry basis)			TOTAL ORGANIC CARBON (TOC) (% by wt. dry basis)		
		A*	P	W	A	P	W	A	P	W	A	P	W	A	P	W	A	P	W	A	P	W
RAW	A	9.1	17.1	15.7	0.22	0.24	0.06	<0.001	<0.001	<0.001	0.22	0.24	0.06	0.12	<0.03	0.014	130	181.0	126	43	23.0	48.0
	B	10.5	†	†	0.11	†	†	0.002	†	†	0.11	†	†	0.10	†	†	120	†	†	39	†	†
PURIFAX																						
Low	A	7.3	16.7	16.0	0.40	0.39	0.07	0.007	0.004	0.005	0.41	0.39	0.08	0.96	0.30	0.41		†			†	
	B	11.0	15.5	12.4	0.31	0.26	0.20	0.006	0.002	0.004	0.32	0.26	0.20	0.66	0.49	0.38						
Medium	A	11.1	18.1	14.1	0.34	0.74	0.31	0.004	0.002	0.006	0.34	0.74	0.32	1.19	0.74	0.43	120.0	105	94.0	59	34.0	39.0
	B	11.1	21.1	20.6	0.58	0.51	0.38	0.003	0.003	0.004	0.58	0.51	0.38	1.15	0.48	0.24	120.0	82	98.0	59	16.0	31.0
High	A	13.5	16.6	16.9	0.40	1.46	0.37	0.001	0.003	0.006	0.40	1.46	0.38	1.25	1.31	1.12	†	208	†	†	36.0	†
	B	7.5	19.7	15.5	1.02	0.33	0.42	0.006	§	0.007	1.03	§	0.43	2.87	0.85	1.27		228			25.0	
HEAT TREATMENT		14.7	21.5	23.2	0.09	0.37	0.02	0.002	<0.001	<0.001	0.09	0.37	0.02	0.10	0.04	0.031	150	§	118	61	§	40.0
DIGESTION†																						
50/50	A	8.52	12.8	14.1	0.03	0.07	0.09	0.004	0.002	<0.001	0.03	0.07	0.09	0.062	0.10	0.04		†			†	
	B	8.52	11.8	11.5	0.25	0.09	0.04	0.003	0.007	<0.001	0.25	0.10	0.04	0.034	0.10	0.05						
75/25	A	10.1	10.9	12.5	0.12	0.07	0.04	0.002	<0.001	<0.001	0.12	0.07	0.04	0.007	0.13	0.05	87	100	100	29	21	16
	B	†	12.1	13.3	†	0.03	0.03	†	0.002	0.002	†	0.03	0.03	†	0.09	0.05	†	110	124	†	38	†
0/100		11.0	12.7	12.2	0.08	0.17	0.09	0.002	<0.001	0.005	0.08	0.17	0.10	0.044	0.06	0.05	†	120	†	†	35	†
SEED		14.4	13.5	11.3	0.34	0.22	0.08	<0.001	<0.002	0.002	0.34	0.22	0.08	0.01	0.07	0.054	76.0	163.0	105	35.0	67.0	32.0

* A and B denote duplicates; A=Amherst; P=Pittsfield; W=Willimantic.
† Fractions indicate ratios of raw sludge/digested sludge seed added to batch digesters.
† Sample was not analyzed.
§ Laboratory error, data lost.

TABLE E2. COMPARISON OF CENTRATES FROM DIFFERENT WASTEWATER PLANTS

SLUDGE SOURCE*		TOTAL SUSPENDED SOLIDS (mg/l)			INSOLUBLE ORGANIC CHLORINE (mg/l)			SOLUBLE ORGANIC CHLORINE (mg/l)			TOTAL ORGANIC CHLORINE (mg/l)			TOTAL INORGANIC CHLORINE (mg/l)			CHEMICAL OXYGEN DEMAND (mg/l)			SOLUBLE CHEMICAL OXYGEN DEMAND (COD)(mg/l)			TOTAL ORGANIC CARBON (TOC) (mg/l)			SOLUBLE TOTAL ORGANIC CARBON (TOC) (mg/l)		
		A*	P	W	A	P	W	A	P	W	A	P	W	A	P	W	A	P	W	A	P	W	A	P	W	A	P	W
RAW	A	540	1420	500	0.22	0.31	1.68	<0.02	0.12	0.04	0.22	0.43	1.72	59	72	55	1680	5760	7640	1410	590	2910	1000	†	3000	690	720	1100
	B	520	†	†	0.21	†	†	0.04	†	†	0.25	†	†	58	†	†	1170	†	†	740	†	†	1100	†	†	800	†	†
PURIFAX																												
Low	A	720	1150	640	3.15	2.7	1.25	0.57	0.52	1.04	3.72	3.2	2.29	860	1210	950	†	†	†	†	†	†	†	†	†	†	†	†
	B	900	2320	460	3.52	3.9	0.63	0.17	0.16	0.42	3.69	4.1	1.05	600	1010	770												
Med.	A	460	930	590	1.68	13.4	1.53	0.93	0.59	0.76	2.61	14.0	2.29	1080	1570	1200	4150	1860	7680	2180	950	3620	1500	†	2800	1000	600	1800
	B	590	1030	530	1.09	10.8	2.21	0.72	0.33	1.16	1.81	11.1	3.37	1010	1550	1280	3120	2130	7420	2410	1020	4370	1800	†	3100	1700	610	1700
High	A	510	600	1290	2.06	4.3	20.8	0.92	0.96	1.00	2.98	5.3	21.8	1340	2650	2470	†	2860	†	†	2130	†	†	†	†	†	760	†
	B	620	740	1690	3.73	14.8	23.2	0.80	0.88	1.55	4.53	15.7	24.8	1900	2430	2040		3190			1910						670	
HEAT TREATMENT		280	74	170	0.31	<0.04	<0.04	0.38	0.08	0.03	0.69	0.04- 0.012	0.03- 0.07	65	86	76	14600		13200	13400		13200	7600		7200	7000		7200
DIGESTION†																												
50/50	A	540	740	100	0.06	0.42	0.03	<0.02	0.13	0.04	0.06- 0.07-	0.08	.55 .31	0.07 0.15- 0.17	76	84	39	†	†	†	†	†	†	†	†	†	†	†	†
	B	800	720	1500	0.70	0.14	0.15	<0.02	0.12	0.02	0.07-	0.72			69	87	69												
75/25	A	710	540	840	0.38	0.12	0.07	0.04	0.10	0.06	0.42	0.22	0.13		61	71	110	650	4360	2010	490	340	1070	380	1100	410	150	130	120
	B	†	1450	600	†	0.19	0.03	†	0.23	0.08	†	0.42	0.11		†	79	41	†	3760	2310	†	810	670	†	1700	650	†	160	140
0/100		320	690	790	0.07	0.12	<0.03	0.03	0.15	0.03	0.10	0.27	0.03-	0.06	58	67	49	†	1580	†	†	840	†	†	960	†	†	360	†
SEED		340	590	110	0.16	<0.04	0.47	<0.02	<0.04	0.02	0.16	<0.08	0.49		32	64	37	§	2710	670	§	360	620	§	§	93	§	140	76

A and B denote duplicates; A=Amherst; P=Pittsfield; W=Willimantic.
Fractions indicate ratios of raw sludge/digested sludge seed added to batch digesters.
Sample was not analyzed.
Laboratory error, data lost.

TABLE E3. MASS BALANCE OF ORGANIC CHLORINE IN SLUDGE STABILIZATION PROCESSES FROM ONE LITER OF AMHERST, MASSACHUSETTS SLUDGE*

		INSOLUBLE ORGANIC CHLORINE		SOLUBLE ORGANIC CHLORINE		RATIO OF TOTAL ORGANIC CHLORINE AFTER PROCESS TO FEED
		CAKE	CENTRATE	CAKE	CENTRATE	
SLUDGE SOURCE †		(mg)	(mg)	(mg)	(mg)	
RAW	A	50	0.1	ND	ND	
	B	25	0.1	0.451	0.3	
PURIFAX						
Low	A	112	2.1	1.968	0.4	1.2 to 4.6
	B	84	2.3	1.627	0.1	0.9 to 3.4
Med.	A	98	1.1	1.147	0.6	2.0 to 3.9
	B	156	0.8	0.806	0.5	3.2 to 6.1
High	A	112	1.5	0.279	0.7	1.2 to 4.4
	B	284	2.8	1.67	0.6	2.9 to 11
HEAT TREATMENT		21	0.3	0.469	0.3	0.9 to 0.4
DIGESTER SEED		74	0.1	ND	ND	
DIGESTION †						
50/50	A	6	0.05	.79	ND	0.1
	B	40	0.6	.48	ND	0.7
75/25	A	27	0.3	.458	0.03	0.4
	B					
0/100		18	0.06	.44	0.02	0.2

* Data reported as milligrams (mg) in sample; (ND) means none detected at reporting limits.

† A and B denote duplicates.

‡ Fractions indicate ratios of raw sludges/digested sludge seed added to digesters.

TABLE E4. MASS BALANCE OF ORGANIC CHLORINE IN SLUDGE STABILIZATION PROCESSES FROM ONE LITER OF PITTSFIELD, MASSACHUSETTS SLUDGE*

SLUDGE SOURCE †		INSOLUBLE ORGANIC CHLORINE CAKE (mg)	INSOLUBLE ORGANIC CHLORINE CENTRATE (mg)	SOLUBLE ORGANIC CHLORINE CAKE (mg)	SOLUBLE ORGANIC CHLORINE CENTRATE (mg)	RATIO OF TOTAL ORGANIC CHLORINE AFTER PROCESS TO FEED
RAW	A	105.53	0.203	ND	0.08	
	B					
PURIFAX						
Low	A	160	1.9	1.6	0.4	1.5
	B	103	2.7	0.8	0.1	1.0
Med.	A	310	9.3	0.8	0.4	3.0
	B	205	7.5	1.2	0.2	2.0
High	A	792	2.6	1.6	0.6	7.5
	B	253	10.5	2.1	0.6	2.5
HEAT TREATMENT		136	ND	ND	0.07	1.3
DIGE[illegible]		68	ND	ND	ND	
D						
		21	0.3	0.6	0.10	0.2
		25	0.1	1.9	0.09	0.3
75/25	A	17	0.1	ND	0.08	0.2
	B	9	0.1	0.6	0.2	0.1
100		36	0.1	ND	0.1	0.3

* Data reported as milligrams (mg) in sample; (ND) means none detected at reporting limits.

† A and B denote duplicates.

‡ Fractions indicate ratios of raw sludges/digested sludge seed added to digesters.

TABLE E5. MASS BALANCE OF ORGANIC CHLORINE IN SLUDGE STABILIZATION PROCESSES FROM ONE LITER OF WILLIMANTIC, MASSACHUSETTS SLUDGE*

		INSOLUBLE ORGANIC CHLORINE		SOLUBLE ORGANIC CHLORINE		RATIO OF TOTAL ORGANIC CHLORINE AFTER PROCESS TO FEED
		CAKE	CENTRATE	CAKE	CENTRATE	
SLUDGE SOURCE †		(mg)	(mg)	(mg)	(mg)	
FEED	A	23	1.0	ND	0.02	
	B					
PURIFAX						
Low	A	23	0.6	1.6	0.5	1.0
	B	65	0.3	1.3	0.2	2.3
Med.	A	105	0.8	2.0	0.4	4.5
	B	118	1.1	1.2	0.6	5.0
High	A	125	11	2.0	0.5	4.9
	B	145	11	2.4	0.8	5.6
HEAT TREATMENT		8	ND	---	0.02	0.3
DIGESTER SEED		20	0.3	0.5	0.01	---
DIGESTION †						
50/50	A	20	0.02	ND	0.03	0.8
	B	11	0.1	ND	ND	0.5
75/25	A	10	0.06	ND	0.05	0.5
	B	7	0.02	0.5	0.07	0.3
100		24	ND	1.3	0.02	1.3

* Data reported as milligrams (mg) in sample; (ND) means none detected at reporting limits.

† A and B denote duplicates.

‡ Fractions indicate ratios of raw sludges/digested sludge seed added to digesters.

TABLE E6. MASS BALANCE OF TOTAL CHLORINE

SLUDGE SOURCE *	Pittsfield mg/l	Pittsfield % Recovery	Amherst mg/l	Amherst % Recovery	Willimantic mg/l	Willimantic % Recovery
RAW A	166	-	115	-	67	-
B			86	-		
SEED	141	-	97	-	62	-
HEAT TREATMENT	198	119	102	101	80	119
DIGESTION †						
25/75 A	114	77	81	83	61	96
B	121		102	104	83	131
50/50 A	107	70	78	79	112	174
B	97	63	‡	‡	52	81
0/100	105	74	76	78	80	129

* A and B denote duplicates.

† Fractions indicate ratios of raw sludges/digested sludge seed added to digesters.

‡ Samples A and B were combined to provide sufficient quantity for analysis.

TABLE E7. ORGANIC PRIORITY POLLUTANTS IN PURIFAX SLUDGE AND CORRESPONDING CAKE AND LIQUID SAMPLE

		506 Purifax-Q Sludge ug/l	507 Purifax Sludge ug/l	500 Purifax-Q Cake ug/kg	501 Purifax Cake ug/kg	513 Purifax Liquid ug/l
101	Cloromethane	19	48	Not Analyzed	Not Analyzed	Not Analyzed
102	Dichlorodifluoromethane	-	-			
105	Chloroethane	21	32			
106	Methylene Chloride	-	-			
112	Trans-1,2-Dichloroethylene	-	-			
113	Chloroform	150	160			
115	1,1,1-Trichloroethane	3	2			
116	Carbon Tetrachloride	170	226			
120	Trichloroethylene	1	1			
121	Benzene	1	1			
127	1,1,2,2-Tetrachloroethylene	4	7			
128	Toluene	13	16			
130	Ethyl Benzene	-	-			
201	2-Chlorophenol	P	P	-	-	P
203	Phenol	P	P	P	P	4
205	2,4 Dichlorophenol	-	11	P	P	P
206	2,4,6-Trichlorophenol	20	26	P	130	4
210	Pentachlorophenol	P	P	P	P	-
301-3	Dichlorobenzenes	12	10	-	170	P
312	1,2,4-Trichlorobenzene	-	-	-	-	-
315	Naphthalene	12	11	120	220	-
324	Fluorene	-	-	-	-	-
326	Diethyl Phthalate	P	P	P	P	P
331-2	Anthracene/Phenanthrene	P	P	P	P	P
333	Di-n-butylphthalate	28	28	400	570	-
334	Fluoranthene	23	23	200	400	P
335	Pyrene	25	25	230	450	P
337	Butyl Benzyl Phthalate	29	28	230	280	P
338-9	Bis(2-ethylhexyl) Phthalate/. Di-noctyl Phthalate	400	350	2,300	3,400	72
340-1	Chrysene/Benzo(a) Anthracene	12	14	120	210	7
343-4	Benzo(b) Fluoranthene/ Benzo(k) Fluoranthene	-	-	-	-	-
345	Benzo(a) Pyrene	-	-	-	-	-
346	Indeno(1,2,3-c,d) Pyrene	-	-	-	P	32

TABLE E7. (Con'td.) ORGANIC PRIORITY POLLUTANTS
IN PURIFAX SLUDGE AND CORRESPONDING CAKE AND LIQUID SAMPLE

		506 Purifax-Q Sludge µg/l	507 Purifax Sludge µg/l	500 Purifax-Q Cake µg/kg	501 Purifax Cake µg/kg	513 Purifax Liquid µg/l
347	Dibenzo(a,h) Anthracene	-	-	-	P	40
348	Benzo(g,h,i) Perylene	-	-	-	-	32
402	Gamma-BHC	-	-	-	-	2
404	Beta-BHC	-	-	-	-	2
417	Chlordane	B	B	-	-	-
419	PCB-1221	C	C	-	-	A
422	PCB-1248	B	C	C	C	-
423	PCB-1254	A		B	B	A
424	PCB-1260	-	B	-	-	-
425	PCB-1016	-		-	-	A

- Not detected

P Detected but below reporting limit

Not Analyzed - No sample was collected for analysis

A 10 to 100 µg/l (µg/kg in the sludge cake)

B 100 to 1000 µg/l (µg/kg in the sludge cake)

C 1000 to 5000 µg/l (µg/kg in the sludge cake)

D 5000 to 10,000 µg/l (µg/kg in the sludge cake)

Q quality control

TABLE E8. ORGANIC PRIORITY POLLUTANTS IN FEED SLUDGE AND CORRESPONDING CAKE SAMPLES

		108 Feed-A Sludge,µg/l	109 Feed Sludge,µg/l	102 Feed-A Cake,µg/kg	103 Feed Cake, µg/kg
101	Chloromethane	-	-		
102	Dichlorodifluoromethane	-	-		
103	Chloroethane	-	-		
104	Methylene Chloride	-	-		
112	Trans-1,2-Dichloroethylene	-	-		
113	Chloroform	19	20		
114	1,1,1-Trichloroethane	1	2		
116	Carbon Tetrachloride				
120	Trichloroethylene	1	2		
121	Benzene	3	3		
127	1,1,2,2-Tetrachloroethylene	2	4		
128	Toluene	16	28		
130	Ethyl Benzene	-	-		
201	2-Chlorophenol	-	-	-	-
203	Phenol	49	47	150	110
205	2,4 Dichlorophenol	P	-	-	P
206	2,4,6-Trichlorophenol	-	-	-	P
210	Pentachlorophenol	P	P	P	120
301-3	Dichlorobenzenes	11	13	70	70
312	1,2,4-Trichlorobenzene	-	-	-	-
313	Naphthalene	12	16	-	120
314	Fluorene	-	-	-	-
316	Diethyl Phthalate	P	-	P	P
318-2	Anthracene/Phenanthrene	P	P	P	P
319	Di-n-butyl Phthalate	23	24	260	240
324	Fluoranthene	22	20	230	210
328	Pyrene	21	23	250	230
337	Butyl Benzyl Phthalate	18	14	200	170
338-9	Bis(2-ethylhexyl) Phthalate/ Di-n-octyl Phthalate	210	230	1,200	1,700
340-1	Chrysene/ Benzo(a) anthracene	14	11	150	140
343-6	Benzo(b) fluoranthene/ Benzo(k) fluoranthene	-	-	74	-
345	Benzo(a) Pyrene	-	-	430	-
348	Indeno (1,2,3-c,d) Pyrene	-	-	750	-
347	Dibenzo(a,h) Anthracene	-	-	430	-
348	Benzo(g,h,i)Perylene	-	-	660	-

TABLE E8. (Con'td). ORGANIC PRIORITY POLLUTANTS
IN FEED SLUDGE AND CORRESPONDING CAKE SAMPLES

		508 Feed-A Sludge,ug/l	509 Feed Sludge,ug/l	502 Feed-A Cake,ug/kg	503 Feed Cake,ug/kg
404	Beta-BHC	-	-	-	36
410	Dieldrin	-	-	28	-
412	DDD	12	-	50	-
414	DDT	190	-	-	-
417	Chlordane	A	A	A	A
419	PCB-1221	-	A	-	B
420	PCB-1232	-	A	A	A
422	PCB-1248	C	-	C	-
423	PCB-1254	B	A	-	B
425	PCB-1016	-	A	-	A

\- Not detected

\# Detected but below reporting limit

Not Analyzed - No sample was collected for analysis

A 10 to 100 ug/l (ug/kg in the sludge cake)

B 100 to 1000 ug/l (ug/kg in the sludge cake)

C 1000 to 5000 ug/l (ug/kg in the sludge cake)

D 5000 to 10,000 ug/l (ug/kg in the sludge cake)

TABLE E9. ORGANIC PRIORITY POLLUTANTS IN SEED SLUDGE AND CORRESPONDING CAKE SAMPLE

		510 Seed Sludge µg/l	504 Seed Cake µg/kg
101	Chloromethane	3	
102	Dichlorodifluoromethane	-	
105	Chloroethane	-	
106	Methylene Chloride	-	
112	Trans-1,2-Dichloroethylene	3	
113	Chloroform	-	
115	1,1,1-Trichloroethane	-	
116	Carbon Tetrachloride	-	
120	Trichloroethylene	-	
121	Benzene	-	
127	1,1,2,2-Tetrachloroethylene	-	
128	Toluene	10	
130	Ethyl Benzene	3	
201	2-Chlorophenol	-	
203	Phenol	P	
205	2,4 Dichlorophenol	-	
206	2,4,6-Trichlorophenol	-	
210	Pentachlorophenol	-	
201-3	Dichlorobenzene	87	360
212	1,2,4-Trichlorobenzene	-	-
213	Naphthalene	20	320
224	Fluorene	-	-
226	Diethyl Phthalate	P	P
231-2	Anthracene/Phenanthrene	22	200
233	Di-n-butyl Phthalate	18	P
234	Fluoranthene	37	300
235	Pyrene	33	330
237	Butyl Benzyl Phthalate	24	400
238-9	Bis(2-ethylhexyl) Phthalate/ Di-octyl Phthalate	300	9,700
240-1	Chrysene/Benzo(a) Anthracene	22	210
243-4	Benzo(b) Fluoranthene/ Benzo(k) Fluoranthene	-	-
245	Benzo(a) Pyrene	-	300
246	Indeno(1,2,3-c,d) Pyrene	P	760
247	Dibenz(a,h) Anthracene	-	400
248	Benzo(g,h,i)Perylene	P	430

TABLE E9. (Con'td). ORGANIC PRIORITY POLLUTANTS
IN SEED SLUDGE AND CORRESPONDING CAKE SAMPLE

		510 Seed Sludge,µg/l	504 Seed Cake,µg/kg
403	Heptachlor	-	39
412	DDD	-	50
417	Chlordane	-	C
422	PCB-1248	B	D
423	PCB-1254	B	D

- Not detected

P Detected but below reporting limit

Not Analyzed - No sample was collected for analysis

A 10 to 100 ug/l (ug/kg in the sludge cake)

B 100 to 1000 ug/l (ug/kg in the sludge cake)

C 1000 to 5000 ug/l (ug/kg in the sludge cake)

D 5000 to 10,000 ug/l (ug/kg in the sludge cake)

TABLE E10. ORGANIC PRIORITY POLLUTANTS IN HEAT TREATED SLUDGE AND CORRESPONDING CAKE SAMPLE

		511 HT Sludge, µg/l	505 HT Cake, µg/kg
101	Chloromethane	-	Not Analyzed
102	Dichlorodifluoromethane	21	
105	Chloroethane	-	
106	Methylene Chloride	-	
112	Trans-1,2-Dichloroethylene	-	
113	Chloroform	-	
115	1,1,1-Trichloroethane	-	
116	Carbon Tetrachloride	-	
120	Trichloroethylene	-	
121	Benzene	11	
127	1,1,2,2-Tetrachloroethylene	-	
128	Toluene	79	
130	Ethyl Benzene	-	
201	2-Chlorophenol	-	-
203	Phenol	27	320
205	2,4 Dichlorophenol	-	-
206	2,4,6-Trichlorophenol	-	-
210	Pentachlorophenol	-	-
301-3	Dichlorobenzene	12	130
312	1,2,4-Trichlorobenzene	-	150
315	Naphthalene	16	190
324	Fluorene	-	P
326	Diethyl Phthalate	-	P
331-2	Anthracene/Phenanthrene	P	P
333	Di-n-butyl Phthalate	P	260
334	Fluoranthene	20	250
335	Pyrene	23	290
337	Butyl Benzyl Phthalate	19	260
338-9	Bis(2-ethylhexyl) Phthalate/ Di-noctylphthalate	340	2,000
340-1	Chrysene/Benzo(a) Anthracene	18	240
343-4	Benzo(b) Fluoranthene/ Benzo(k) Fluoranthene	-	-
345	Benzo(a) Pyrene	-	-
346	Indeno(1,2,3-c,d) Pyrene	-	-
347	Dibenzo (a,h) Anthracene	-	630
348	Benzo(g,h,i) Perylene	-	-
409	DDE	-	26
417	Chlordane	A	-
423	PCB-1254	-	B

- Not detected

P Detected but below reporting limit

Not analyzed - No sample was collected for analysis

A 10 to 100 µg/l (µg/kg in the sludge cake)

B 100 to 1000 µg/l (µg/kg in the sludge cake)

TABLE E11. ORGANIC PRIORITY POLLUTANTS IN DIGESTED SLUDGE AND CORRESPONDING CAKE SAMPLE

		514 Sludge, µg/l	515 Cake, µg/kg
101	Chloromethane	-	Not Analyzed
102	Dichlorodifluoromethane	-	
105	Chloroethane	-	
106	Methylene Chloride	99	
112	Trans-1,2- Dichloroethylene	-	
113	Chloroform	-	
115	1,1,1-Trichloroethane	32	
116	Carbon Tetrachloride	-	
120	Trichloroethylene	-	
121	Benzene	-	
127	1,1,2,2-Tetrachloroethylene	-	
128	Toluene	-	
130	Ethyl Benzene	-	
201	2-Chlorophenol	-	-
203	Phenol	P	P
205	2,4 Dichlorophenol	-	-
206	2,4,6-Trichlorophenol	-	-
210	Pentachlorophenol	-	-
301-3	Dichlorobenzene	54	-
312	1,2,4-Trichlorobenzene	-	-
315	Naphthalene	34	260
324	Fluorene	-	-
326	Diethyl Phthalate	-	P
331-2	Anthracene/Phenanthrene	P	P
333	Di-n-butyl Phthalate	22	-
334	Fluoranthene	64	430
335	Pyrene	58	-
337	Butyl Benzyl Phthalate	34	-
338-9	Bis(2-ethylhexyl) Phthalate/ Di-noctyl Phthalate	840	5,300
340-1	Chrysene/Benzo(a) Anthracene	35	240
343-4	Benzo(b) Fluoranthene/ Benzo(k) Fluoranthene	-	-
345	Benzo(a) Pyrene	-	-
346	Indeno(1,2,3-c,d) Pyrene	160	-
347	Dibenzo(a,h) Anthracene	-	-
348	Benzo(g,h,i) Perylene	140	-
421	PCB-1242	A	-
422	PCB-1248	-	A

- Not detected

P Detected but below reporting limit

Not Analyzed - No sample was collected for analysis

A 10 to 100 µg/l (µg/kg in the sludge cake)

TABLE E12. ORGANIC PRIORITY POLLUTANTS NEVER DETECTED IN SLUDGE, CAKE OR LIQUID SAMPLES

103.	Bromomethane
104.	Vinyl Chloride
107.	Acrolein
108.	Trichlorofluoromethane
109.	Acrylonitrile
110.	1,1-Dichloroethylene
111.	1,1-Dichloroethane
114.	1,2-Dichloroethane
117.	Bromodichloromethane
118.	1,2-Dichloropropane
119.	Trans-1,3-Dichloropropylene
122.	Cis-1,3-Dichloropropylene
123.	Dibromochloromethane
124.	1,1,2-Trichloroethane
125.	Bromoform
126	1,1,2,2-Tetrachloroethane
129.	Chlorobenzene
202.	2-Nitrophenol
204.	2,4-Dimethylphenol
207.	4-Chloro-3-Cresol
208.	2,4-Dinitrophenol
209.	4,6-Dinitro-2-Cresol
211.	4-Nitrophenol
304.	Hexachloroethane
305.	Bis(chloromethyl) Ether
306.	Bis(2-chloromethyl) Ether
307.	Bis(2-chloroisopropyl) Ether
308.	N-Nitrosodimethylamine
309.	Nitrosodi-n-propylamine
310.	Nitrobenzene
311.	Hexachlorobutadiene
313.	2-Chloroethyl Vinyl Ether
314.	Bis(2-chloroethoxy) Methane
316.	Isophorone
317.	Hexachlorocyclopentadiene
318.	2-chloronaphthalene
319.	Acenaphthylene
320.	Acenaphthene
321.	Dimethyl Phthalate
322.	2,6-Dinitrotoluene
323.	4-Chlorophenyl Phenyl Ether
325.	2,4-Dinitrotoluene
327.	1,2-Diphenylhydrazine
328.	N-Nitrosodiphenylamine
329.	Hexachlorobenzene
330.	4-Bromophenyl Phenyl Ether
336.	Benzidine
342.	3,3-Dichlorobenzidine
401.	Alpha-BHC
405.	Delta-BHC
406.	Aldrin
407.	Heptachlor Epoxide
408.	Endosulfan I
411.	Endrin
413.	Endosulfan II
415.	Endrin Aldehyde
416.	Endosulfan Sulfate
418.	Toxaphene

TABLE E13. SUMMARY OF RESULTS FOR PPINICHI ANALYSIS OF SAMPLE EXTRACTS

Sample	Sample Code	Fig. No.	Chromatographic Peaks? (+)CI*	(-)CI	M.S. Scan No. of Chlorinated Peak(s)	Identity
Purifax Sludge	A-500	A1	+	+	1395	
	B-500	A2	+	+	824,873,965,975,1016 1107,1158,1263,1435,1384	
Purifax Sludge	A-501	A3	+	+	576,1407	chloroacetophenone or chloropropylbenzene?
	B-501	A4	-	-	-	
Feed Sludge	B-502	A5	+	+	1401,1618	
	B-502	A6	+	+	832,1391	
Feed Sludge	A-503	A7	+	+	1400	
	B-503	A8	-	-	-	
Digester Seed Sludge	A-504	A9	+	+	412,1390	
	B-504	A10	+	+	1381,1432	
Heat Treatment Sludge	A-505	A11	+	+	570,1272,1426	
	B-505	A12	+	+	1382	
Purifax Cake	A-506	A13	+	+	273,247,580,884 970,986,1166	273-tetrachlorobutane 247-trichloromethylpropene 580-chloroacetophenone or chloropropylbenzene
	B-506	A14	+	+	240,261,569,694,762, 773,971,1069,1170,1385	240-chlorophenol
Purifax Cake	A-507	A15	+	+	248,271,578,611,709,829, 878,935,985,1023,1079 1270	248-trichloromethylpropene 271-tetrachlorobutane 578-chloroacetophenone or chloropropylbenzene
	B-507	A16	+	+	262,330,570,694,974,1242, 1388	

(continued)

TABLE E13. SUMMARY OF RESULTS FOR PPINICHI ANALYSIS OF SAMPLE EXTRACTS
(continued)

Sample	Sample Code	Fig. No.	Chromatographic Peaks? (+)CI*	Chromatographic Peaks? (-)CI	M.S. Scan No. of Chlorinated Peak(s)	Identity
Feed	A-508	A17	+	+	290,1458	
Cake	B-508	A18	+	+	1387	
Feed	A-509	A19	+	+	281,398,1437,2669	
Cake	B-509	A20	+	+	1386	
Digester Seed	A-510	A21	+	+	276,397,1420,1440,2551, 2601	
Cake	B-510	A22	+	+	1390	
Heat treatment Cake	A-511	A23	+	+	757,1530	
	B-511	A24	+	+	1397	
Blank	A-512	A25	-	+	-	
	B-512	A26	-	-	-	
Purifax Centrate	A-513	A27	+	+	241,266,577,884,1170,1418	
	B-513	A28	+	+	262,1394	
Digested Sludge	A-514	A29	+	+	283,386,1446,2062,2170, 2664	
	B-514	A30	+	+	1389	
Digested Cake	A-515	A31	-	+	355	
	B-515	A32	+	+	1394	

* CI = chemical ionization mode.

TABLE E14. SUMMARY OF RESULTS OBTAINED FOR EXTRACTS FROM LEACHATE SAMPLES USING PPINICHI ANALYSIS

Sample	Sample Code	Fig. No.	Chromatographic Peaks? (+)CI*	(-)CI	M.S. Scan No. of Chlorinated Peak(s)	Identity
Digested	6960-Acid	A33	-	-	-	
	6960-Base	A34	+	+	-	
Purifax	6961-Acid	A35	+	+		
	6961-Base	A36	+	+	-	
Purifax	6962-Acid	A37	+	+		Chloroacetophenone or chloropropylbenzene
	6962-Base	A38	-	+	-	
Digester	6693-Acid	A39	-	-	-	
Seed	6693-Base	A40	+	+	-	
Feed	6694-Acid	A41	+	+	-	
	6694-Base	A42	+	+	-	
Feed	6965-Acid	A43	+	-	-	
	6965-Base	A44	+	+	-	
Digester	AH-0 B+N-Acid	A45	-	-	-	
	AH-0 B+N-Base	A46	-	-	-	

* CI = chemical ionization mode.

TABLE E15. SUMMARY OF QUALITATIVE CONFIRMATION OF A. D. LITTLE HALOGENATED EXTRACTABLE PRIORITY POLLUTANT ANALYSIS

Sample Code	Compounds	NI/PI/CI Confirmation	
		Acid Extract	Base Extract
500	Dichlorophenol	(-)	(-)
Purifax	Trichlorophenol	(-)	(-)
Sludge	Pentachlorophenol	(-)	(-)
	PCB	(+)	(+)
501	Dichlorophenol	(-)	(-)
Purifax	Trichlorophenol	(-)	(-)
Sludge	Pentachlorophenol	(-)	(-)
	Dichlorobenzene	(-)	(-)
	PCB	(+)	(-)
502	Pentachlorophenol	(-)	(-)
Feed	Dichlorobenzene	(-)	(-)
Sludge	Hexachlorobenzene	(-)	(-)
	Chlordane	(-)	(-)
	PCB	(+)	(-)
	Dieldrin	(-)	(-)
	DDD	(-)	(-)
503	Dichlorophenol	(-)	(-)
Feed	Trichlorophenol	(-)	(-)
Sludge	Pentachlorophenol	(-)	(-)
	Dichlorobenzene	(-)	(-)
	Hexachlorobenzene	(-)	(-)
	Chlordane	(-)	(-)
	PCB	(+)	(-)
504	Heptachlor	(-)	(-)
Digester	Dichlorobenzene	(-)	(-)
Seed	Chlordane	(-)	(-)
Sludge	PCB	(+)	(+)
	DDD	(-)	(-)
505	Dichlorobenzene	(-)	(-)
Heat	Trichlorobenzene	(+)	(-)
Treatment	PCB	(+)	(+)
Sludge	DDE	(-)	(-)
506	Chlorophenol	(-)	(+)
Purifax	Trichlorophenol	(-)	(-)
Cake	Pentachlorophenol	(-)	(-)
	Dichlorobenzene	(-)	(-)
	Chlordane	(-)	(-)
	PCB	(-)	(-)

TABLE E15. (Cont.) SUMMARY OF QUALITATIVE CONFIRMATION OF A. D. LITTLE HALOGENATED EXTRACTABLE PRIORITY POLLUTANT ANALYSIS

Sample Code	Compounds	NI/PI/CI Confirmation Acid Extract	Base Extract
507 Purifax Cake	Chlorophenol	(-)	(-)
	Dichlorophenol	(-)	(-)
	Trichlorophenol	(-)	(-)
	Pentachlorophenol	(-)	(-)
	Dichlorobenzene	(-)	(-)
	Chlordane	(-)	(-)
	PCB	(+)	(+)
508 Feed Cake	Dichlorophenol	(-)	(-)
	Pentachlorophenol	(-)	(-)
	Dichlorobenzene	(-)	(-)
	Hexachlorobenzene	(-)	(-)
	Chlordane	(-)	(-)
	PCB	(+)	(+)
509 Feed Cake	Pentachlorophenol	(-)	(-)
	Dichlorobenzene	(-)	(-)
	Hexachlorobenzene	(-)	(-)
	Chlordane	(-)	(-)
	PCB	(+)	(+)
510 Digester Seed Cake	Dichlorobenzene	(-)	(-)
	PCB	(+)	(+)
511 Heat Treatment Cake	Dichlorobenzene	(-)	(-)
	Chlordane	(+)	(+)
513 Purifax Centrate	Chlorophenol	(-)	(-)
	Dichlorophenol	(-)	(-)
	Trichlorophenol	(-)	(-)
	Dichlorobenzene	(-)	(-)
	Hexachlorobenzene	(-)	(-)
	PCB	(+)	(+)
514 Digested Sludge	Dichlorobenzene	(-)	(-)
	PCB	(+)	(+)
515 Digested Cake	PCB	(+)	(+)

ture of polychlorinated biphenyl compounds containing from one to ten chlorine atoms. Further, two of the pesticides, chlordane and toxaphene, are not just one compound.

Identification of pesticides and PCBs by gas chromatography/electron capture detection (GC/ECD) is based on matching the retention time of the compound (time from the start of the run) relative to that of an internal standard, which is added to each sample. In addition, for PCB identification, the relative abundance pattern of the GC peaks must match that obtained from reference chromatograms. Many of the PCBs have GC peaks in common. The relative amounts of each peak, as indicated by the peak heights, define the pattern for each of the PCBs; however, the overlap of several PCBs makes mixture specific assignments difficult. In addition, analytical difficulty prohibits making unequivocal assignments of the PCBs in the sludge and cake samples. The samples were very complex and required extensive cleaning before analysis by GC/ECD according to the priority pollutant protocol. The extra cleanup steps required more effort than normal for this particular analysis.

The chromatograms were examined very carefully for PCB identification. The assignments arrived at were made only after the peak relative retention times and peak abundunces and profiles matched exactly. There appeared, however, to be several PCBs in many of these samples, and as such, it was not possible to reliably differentiate between the. Because of this uncertainty in specific PCB assignment (e.g., PCB 1248 vs. PCB 1260), it also seemed possible (but highly unlikely) that many of the peaks were due to nonpesticide or PCB species.

ADL observed that four different PCBs could possibly be present in the seed sludge sample (No. 510): PCB 1232, 1248, and 1254. Since these four PCBs have specific chlorinated byphenyl isomers in common, none of the GC peaks observed could be attributed to only one PCB mixture.

Due to this overlapping, it is very difficult to conclude with certainty which individual PCBs are present and, therefore, to accurately estimate the level of any individual PCB. Further, these samples have been exposed to field conditions (i.e., weathering) which could modify the pattern of GC peaks that are observed for a single PCB in comparison to the original PCB, making the identification an even more difficult task.

CHLORINATED ORGANICS

Table E13 presents a summary of the results for the PPINICH analysis of sample extracts. The table indicates whether chromatographic peaks were detected in the positive and/or negative ion chemical ionization modes and the mass spectral scan number of the chlorinated peak in each of the chromatographic runs. The identity of some compounds are indicated. Only a small fraction of the chlorinated peaks was identified. Table E14 gives a summary of results for extracts from leachates samples using the same technique.

Because the PPINICHI method confirmed few of the priority pollutants, the data were further analyzed. Table E15 lists the chlorinated priority pollutants

found and indicates whether the presence of the compound was confirmed.

A negative (-) sign indicates that the compound was not confirmed; a positive (+) sign indicates that the compound was confirmed. Few of the results could be confirmed. By contrast, PCBs were identified and confirmed in all samples except one: Purifax sludge cake (506). Examination of the data indicated chlorinated compounds with retention times similar to compounds reported by ADL were present, but their identity was different.

APPENDIX F

STATISTICAL ANALYSIS OF RESULTS

A series of tests was conducted to assess the reliability of the data in terms of laboratory error and sludge treatment error. Amherst sludge was used for this test because Amherst is a domestic plant, and, as such, the results would not be influenced by an industrial waste fraction. This sludge was also used in the tests conducted by RTI and ADL for identification of chlorinated compounds.

Testing was conducted only with Purifax tests aimed at achieving a medium concentration. The purpose of the test was to assess the variability of results of the entire study process from sample mixing through processing to analysis. The test procedure was almost identical to the procedure for earlier testing. Otherwise, the results could not be compared statistically.

The test was conducted as follows: 75 liters of sludge were mixed and divided for testing by the Purifax process and for chemical analysis of feed samples. Six Purifax runs were conducted in all. Three tests were conducted at about 10°C; the other three were tested at about 18°C. In earlier tests, temperature had been recorded, but not controlled. In these tests, temperature was controlled to see its effect on the tests results (see Table F1 for test data). As described in Appendix B, each of the six 2.5-liter Purifax samples was broken into four portions before centrifuging. The resultant four cakes and four centrates were then carefully mixed to produce one cake and one centrate sample. These samples were each divided into three equal portions for replicate analysis.

Results of the analyses are shown in Table F2. Most of the results are consistent; however, there are some that seem questionable when compared to neighboring results. Computations were rechecked, but no errors were found.

Observations

The third centrate replicate from the fourth Purifax run contained a much higher concentration of insoluble organic chlorine than the other two replicates. It was also observed that this third replicate also contained much more suspended solids than the other two. The suspended solids were then analyzed in all three centrates, and the third replicate was re-analyzed for insoluble organic chlorine after allowing the solids to settle for 90 minutes.

Table F3 shows that the concentration of insoluble organic chlorine in the centrate dropped dramatically after settling. So, it is likely that variations in suspended solids levels are responsible for the original dif-

TABLE F1. TEST DATA FOR RELIABILITY STUDY

Run No.	Time	Raw Sludge pH	Raw Sludge % Solids	Raw Sludge Color	Raw Sludge °C	Preload (ml)*	Chlorine Application (mg/l) Dosage+	Residual	Demand	Purifax Treated Sludge Treat. Period (min)	pH	Color	°C	Comments
1	12:40 pm	5.5	4.0	Dark Grey	17	900	2,500	240	2,260	10	2.5	Light Brown	23	Cl_2 and vacuum valves were leaking; replaced
2	1:10	5.5	40	"	18	1,000	2,300	210	2,090	10	2.7	Medium Brown	24	
3	1:30	5.5	4.0	"	18	1,200	1,900	170	1,730	10	3.1	Grayish Brown	23	
4	2:20	5.35	4.0	"	9	900	2,500	260	2,240	10	2.45	Light Brown	19	
5	2:55	5.35	4.0	"	10	1,000	2,300	215	2,085	10	2.65	Medium Brown	21	
6	3:20	5.35	4.0	"	11	1,200	1,900	170	1,730	10	3.1	Medium Brown	19	

*Quantity initially pumped into reaction chamber.

+From BIF chart based on preload.

TABLE F2. ORGANIC CHLORINE AND SOLIDS DATA FROM RELIABILITY STUDY

Sample Description	Volume of Cake (ml)/1000 ml of sample	Insoluble Organic Chlorine		Soluble Organic Chlorine		Total Organic Chlorine		Total Solids	
		mg/l	% by wt. dry basis	mg/l	% by wt. dry basis	mg/l	% by wt. dry basis	mg/l	% by wt. dry basis
FEED SLUDGE	--	--	<0.04	--	0.001	--	0.001-0.04	29,500	3.21
CAKE	290	--	<0.02	--	0.001	--	0.001-0.02	--	12.3
		--	<0.02	--	0.004	--	0.004-0.02	--	12.7
		--	0.10	--	0.001	--	0.10	--	14.3
CENTRATE		<0.02	--	0.04	--	0.04-0.06	--	--	--
		0.05	--	0.06	--	0.11	--	--	--
		0.03	--	0.02	--	0.02-0.05	--	--	--
1st PURIFAX RUN SLUDGE		--	0.54	--	0.005	--	0.55	37,300	3.83
CAKE	295	--	0.66	--	0.003	--	0.66	--	12.6
		--	0.33	--	0.003	--	0.33	--	14.8
		--	0.58	--	0.004	--	0.58	--	14.1
CENTRATE		12.7	--	0.59	--	13.3	--	--	--
		7.5	--	0.52	--	8.0	--	--	--
		4.2	--	0.53	--	4.7	--	--	--
2nd PURIFAX RUN SLUDGE		--	0.51	--	0.004	--	0.51	38,000	3.98
CAKE	300	--	0.46	--	0.003	--	0.46	--	12.8
		--	0.52	--	0.005	--	0.53	--	14.8
		--	0.42	--	0.003	--	0.42	--	18.0
CENTRATE		3.4	--	0.46	--	3.9	--	--	--
		5.2	--	0.46	--	5.7	--	--	--
		5.2	--	0.40	--	5.6	--	--	--
3rd PURIFAX RUN SLUDGE		--	0.46	--	0.003	--	0.46	37,500	3.96
CAKE	285	--	0.11	--	0.003	--	0.11	--	16.1
		--	0.54	--	0.006	--	0.55	--	12.1
		--	0.18	--	0.004	--	0.18	--	14.5
CENTRATE		5.3	--	0.40	--	5.7	--	--	--
		2.9	--	0.32	--	3.2	--	--	--
		3.3	--	0.35	--	3.7	--	--	--

(continued)

TABLE F2. ORGANIC CHLORINE AND SOLIDS DATA FROM RELIABILITY STUDY
(continued)

Sample Description	Volume of Cake (ml)/1000 ml of sample	Insoluble Organic Chlorine		Soluble Organic Chlorine		Total Organic Chlorine		Total Solids	
		mg/l	% by wt. dry basis	mg/l	% by wt. dry basis	mg/l	% by wt. dry basis	mg/l	% by wt. dry basis
4th PURIFAX RUN SLUDGE		--	0.44	--	0.003	--	0.44	38,700	4.01
CAKE	300	--	0.31	--	0.003	--	0.31	--	15.5
		--	0.53	--	0.003	--	0.53	--	14.7
		--	0.69	--	0.003	--	0.69	--	12.8
CENTRATE		7.7	--	0.56	--	8.3	--	--	--
		2.4	--	0.37	--	2.8	--	--	--
		69.3	--	0.70	--	70.0	--	--	--
5th PURIFAX RUN SLUDGE		--	0.57	--	0.003	--	0.57	38,300	3.99
CAKE	290	--	0.43	--	0.004	--	0.43	--	16.6
		--	0.42	--	0.003	--	0.42	--	16.8
			0.62		0.003		0.62		15.9
CENTRATE		19.7	--	0.47	--	20.2	--	--	--
		27.0	--	0.40	--	27.4	--	--	--
		20.1	--	0.42	--	20.5	--	--	--
6th PURIFAX RUN SLUDGE		--	0.47	--	0.003	--	0.47	36,100	3.78
CAKE	310	--	0.21	--	0.003	--	0.21	--	13.8
		--	0.14	--	0.004	--	0.14	--	13.7
		--	0.21	--	0.003	--	0.21	--	16.5
CENTRATE		11.0	--	0.38	--	11.4	--	--	--
		14.2	--	0.60	--	14.8	--	--	--
		9.0		0.53		9.5	--	--	--

TABLE F3. REVIEW OF DATA FOR CENTRATE FROM FOURTH RUN

Description	Suspended Solids (mg/l)	Soluble Organic Chlorine, mg/l	Insoluble Organic Chlorine, mg/l
1st replicate	980	0.56	7.7
2nd replicate	340	0.37	2.4
3rd replicate	2,100	0.70	69.3
		(After settling)	5.4

ference in insoluble organic chlorine results.

The data from the pilot tests suggest that the Purifax process produces higher concentrations of chlorinated organics than digestion and heat conditoning do. To test the statistical significance of the findings, the results of the reliability tests were analyzed and applied to the pilot data.

Because the cake chlorine levels are an order of magnitude larger than in centrate, cake chlorine levels were used in investigating the statistical significance of the tests. In addition, analytical reliability for the cake was much better than for centrate. Experimental errors for the centrate samples were of the same order as concentrations.

Table F4 shows insoluble chlorines in the cake from each of the six runs (3 analyses per run). The first three were run at about 18°C; the other three were run at about 10°C. Using the d test and the t test (Crow et al., 1960)* we determined that the insoluble inorganic chlorine in the cake resulting from testing at the two different temperatures have statistically the same mean. Hence, it is safe to use the data from the two temperatures together. Within each temperature, however, the mean value for insoluble inorganic chlorine decreased with decreased chlorine residual. This observation questions the validity of lumping data for analysis, although all residuals and dosages are in or near the range defining "medium" applications. Lumping is nevertheless necessary to allow any type of analysis. It may be reasonably justified given the reliability of variations of dosage in practice.

For the Purifax results to be statistically different from others, it must be shown that concentrations in other samples lie outside the range of results that would be reasonably expected from sludge that has received processing by the Purifax process. If the results fall within a reasonable range, then they would not be significantly different.

* Crow, E. L., S. A. Davis, and M. W. Maxfield. Statistics Manual. Dover, New York. 1960.

TABLE F4. INSOLUBLE ORGANIC CHLORINE IN CAKES (RELIABILITY STUDY)

Sample Type	Sample Temperature (°C)	Chlorine Residual (mg/l)	Results (mg/l)	Mean (mg/l)	Variance (mg/l)	Coefficient of Variation
1st run	17	240	245.32 144.08 241.25	210.22	3284.46	0.27
2nd run	18	210	176.64 230.88 218.40	208.64	807.13	0.14
3rd run	18	170	50.47 186.22 74.39	103.69	5250.45	0.70
4th run	9	260	144.15 196.02 300.15	213.44	6312.30	0.37
5th run	10	215	207.00 204.62 285.88	232.5	2138.14	0.20
6th run	11	170	89.84 59.46 107.42	85.57	588.55	0.28

* mg/l refers to mass of chlorine in cake divided by volume of portion centrifuged to produce cake.

The logic used for this investigation is shown in Figure F1. The bell-shaped curve is a hypothetical probability density function for insoluble organic chlorine in a cake that has received Purifax processing. The shaded area of the distribution function indicates the probability that the actual value of the Purifax test result would be less than or equal to the indicated value for the non-Purifax sample. If the area is small, then there would be little likelihood that the true value for a Purifax test would be less than that for the non-Purifax sample. If the area is large, then the chance that there would be no difference between the test results would increase. We used two methods to estimate the shaded area shown in the figure. One method, which uses the Tchebychev inequality, makes no assumption about the sample of the distribution function. The other method assumes that the distribution is unimodal, with rapidly decaying trails. The normal distribution fits this shape, but many other do too.

With the Tchebychev inequality, the probability that samples come from the same population (i.e., that results from Purifax and non-Purifax samples are statistically the same) is equal to or greater than

$$1 - \frac{1}{h^2} \quad \text{where}$$

h is the number of standard deviation between the concentration in the non-Purifax sample and the mean of Purifax samples. According to Benjamin and Cornell (1970)*, the probability for one model with a unimodal assumption is equal to or greater than

$$1 - \frac{1}{2.25h^2}$$

The Tchebychev inequality cannot be used with h less than 1 (in other words, for results differing by less than one standard deviation from the mean) because h less than 1 produces negative numbers in the equation. The unimodal assumption cannot be used with h less than two-thirds.

With the Tchebychev inequality, the area of the shaded portion is less than or equal to $\frac{1}{2h^2}$. With the unimodal assumption, the shaded area is less than or equal to $\frac{1}{4.5h^2}$.

To apply these two formulas to the results of the analyses for the three treatment plant sludges, we assumed that the coefficient of variation for all sludges treated by the Purifax process was 0.43, the same as for the reliability tests. (The coefficient of variation is the standard deviation divided by the sample mean.) Then, we calculated the standard deviations for the pilot results by multiplying their mean by 0.43. Next, we used these estimates to determine the area of the shaded portion -- the probability that the non-Purifax sample did not come from the same population as the Purifax sample. Results are shown on Table F5.

The left-hand column lists the source of the sludge and the treatment that it received. The other three columns are headed by high, medium, or low to denote the residual from the Purifax treatment that the left-hand column is being compared with. The values in the columns are the probability that the process listed in the left-hand column produces the same concentration of chlorine in the processed sludge as the Purifax treatment being compared. There are two values shown for each combination: the left-hand value -- the lower one -- assumes a unimodal distribution; the right-hand value makes no assumption regarding the distribution function.

According to Table F-5, the probability that Purifax treatment produces the same concentrations of insoluble organic chlorine in cake as other processes

* Benjamin, J.R., and C.A. Cornell. Probability, Statistics and Decision for Civil Engineers. McGraw-Hill, New York, 1970.

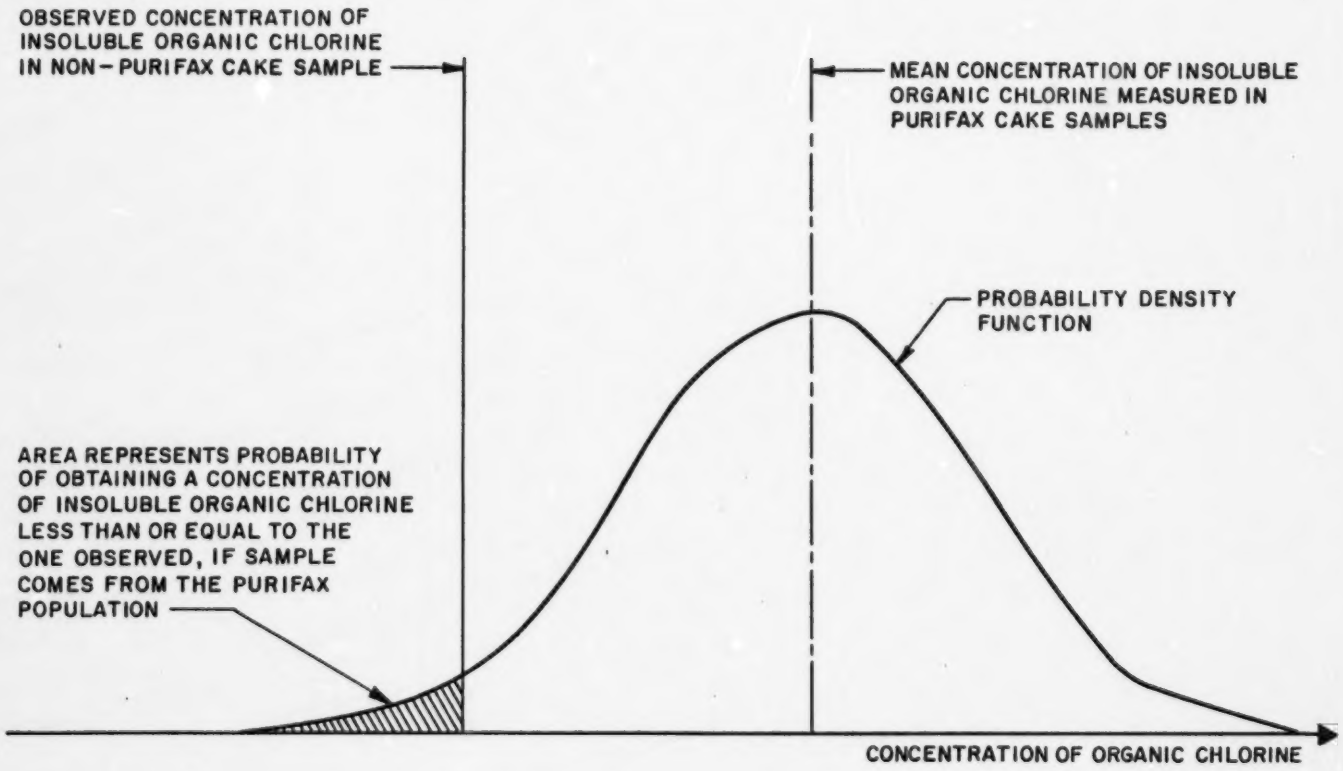
OBSERVED CONCENTRATION OF INSOLUBLE ORGANIC CHLORINE IN NON-PURIFAX CAKE SAMPLE
MEAN CONCENTRATION OF INSOLUBLE ORGANIC CHLORINE MEASURED IN PURIFAX CAKE SAMPLES
PROBABILITY DENSITY FUNCTION
AREA REPRESENTS PROBABILITY OF OBTAINING A CONCENTRATION OF INSOLUBLE ORGANIC CHLORINE LESS THAN OR EQUAL TO THE ONE OBSERVED, IF SAMPLE COMES FROM THE PURIFAX POPULATION
CONCENTRATION OF ORGANIC CHLORINE

FIGURE FI. LOGIC FOR STATISTICAL ANALYSIS

is less than 22.5 percent with the Tchebychev inequality (or 10 percent with the unimodal assumption), except for three samples.

The Pittsfield sludge with low-dosage Purifax treatment was determined to have less organic chlorine than heat conditioned sludge. Also, the difference between the low-dosage Purifax and the sludge feed for Pittsfield was less than two-thirds of a standard deviation. So, neither inequality applies.

The table also shows that statistically the probability is less than 29.6 or 66.5 percent (depending on assumptions) that low-dosage Purifax treatment of Willimantic sludge produced the same concentration of insoluble organic chlorine in cake as in the cake from the feed sludge.

TABLE F5. THE PROBABILITY THAT THE CONCENTRATION OF INSOLUBLE ORGANIC CHLORINE IN PURIFAX CAKES IS THE SAME AS IN OTHER SAMPLES (PERCENT)

Sludge Source and Treatment	Level of Purifax Treatment		
Amherst	High	Medium	Low
HEAT CONDITIONED	≤5.2 - ≤11.6†	≤6.1 - ≤13.7	≤6.6 - ≤15
DIGESTED 50/50*	≤5.2 - ≤11.8	≤6.4 - ≤14.3	≤7 - ≤15.8
75/25	≤5.5 - ≤12.4	≤6.8 - ≤15.4	≤7.8 - ≤17.5
UNTREATED FEED	≤6.2 - ≤14.1	≤8.5 - ≤19.1	≤10.6 - ≤23.8
Willimantic			
HEAT CONDITIONED	≤4.4 - ≤9.9	≤4.8 - ≤10.8	≤6.3 - ≤14.3
DIGESTED 50/50	≤5.2 - ≤11.8	≤5.6 - ≤12.6	≤10 - ≤22.5
75/25	≤4.7 - ≤10.6	≤4.8 - ≤10.9	≤6.4 - ≤14.5
UNTREATED FEED	≤6.5 - ≤14.6	≤7.2 - ≤16.3	≤29.6 - ≤66.5
Pittsfield			
HEAT CONDITIONED	≤7.5 - ≤16.9	≤18.5 - ≤41.5	**
DIGESTED 50/50	≤4.5 - ≤10.1	≤4.9 - ≤11.1	≤6.0 - ≤13.6
75/25	≤4.3 - ≤9.7	≤4.5 - ≤10.2	≤5.0 - ≤11.4
UNTREATED FEED	≤6.4 - ≤14.6	≤11.8 - ≤26.5	**

* 50/50 denotes 50 percent raw sludge and 50 percent digester seed fed to batch digester.

† Left-hand values assumed unimodal distribution. High values make no assumption regarding distribution.

** See text (statistical model invalid for these cases).